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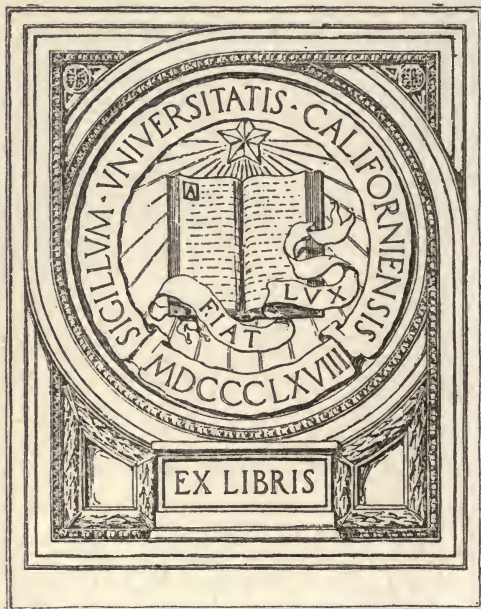


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LABORATORY EXPERIMENTS.

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NO. VIII
ANNALS

LABORATORY EXPERIMENTS

IN

GENERAL CHEMISTRY.

*COMPILED FROM ELIOT AND STORER'S MANUAL
AND OTHER SOURCES,*

BY

WM. RIPLEY NICHOLS AND LEWIS M. NORTON

FOR THE USE OF STUDENTS OF THE MASSACHUSETTS
INSTITUTE OF TECHNOLOGY.

REVISED BY FRED L. BARDWELL.

BOSTON, MASS.

1891.

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GIFT OF Edmund O'Neill

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INTRODUCTION.

As this pamphlet may fall into the hands of teachers and others outside of the Institute, it may be proper to say a few words by way of introduction.

The pamphlet is not intended to be used without a teacher.

As far as possible the directions are given which will enable the student to perform the experiments successfully, but he is left to make his own observations, and then to interpret the results, with such aid as may be necessary from the instructors who are always present in the laboratory. This system, which was introduced by Professor Caldwell in his "Introductory Chemical Practice," requires a careful inspection of the laboratory note-books.

At the Institute most of the experiments are performed on the lecture table before they are attempted by the student, and any text-book may be consulted outside the laboratory; in the laboratory itself no other book than this is allowed. In some cases a sketch of the necessary apparatus, or an example of the apparatus itself, is placed in the laboratory, where it can be seen by the students.



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TO THE STUDENT.

UNLESS special directions to the contrary are given, no text-book will be allowed in the laboratory, except this pamphlet.

The laboratory work should teach us:

- (1) To *observe* and to distinguish essential from non-essential phenomena;
- (2) To express in writing the results of observation;
- (3) To draw proper conclusions as to what facts are taught by the experiments.

The student's standing is largely determined by the quality of his laboratory work and note-book. The quality is more important than the quantity, and one experiment well and intelligently performed is worth more than a dozen, or, indeed, than any number of experiments performed mechanically without intelligence. The notes must be written clearly and distinctly, in the laboratory note-books. As a rule answer concisely, but fully, the following questions:

- (1) What materials did you use?
- (2) What apparatus did you use?
- (3) What did you do?
- (4) What did you observe?
- (5) What has the experiment taught you?

Begin the entry for each day with the date. Do not crowd your notes. Leave room for remarks, additions, or corrections. Allow a paragraph for each one of the above headings.

Make the record of an experiment as soon as the experiment is performed, and present the notes to an instructor for examination before doing other work.

It is not expected that *all* the experiments in a given set will be performed at any one exercise by all the students, but each student will do only as many of the experiments in the set as he can do well. Leave the note-book on the outside of the desk at the close of each exercise.

At the last exercise in each term the desks should be left in good order. No allowance is made for apparatus which is not in proper condition to be served out to other students. No allowance is made for files, iron pincers, filters, or bits of tubing.

I have read the above directions carefully, and endeavored to understand them.

(Name here.)

MASSACHUSETTS INSTITUTE OF TECHNOLOGY.

First Year.

GENERAL CHEMISTRY.

EXERCISE I. — PRELIMINARY WORK.

1. Ascertain the number of your desk.
2. Find coat-hook corresponding to your desk.
3. Obtain key of desk at the supply room.
4. Examine contents of desk.
5. Hand in the receipt at the supply room.
6. Take the piece of ignition tubing in your desk and report to one of the instructors.

Experiment 1. Mix thoroughly 3 grams of coarsely powdered sulphur with 8 grams of copper filings or fine turnings. Put the mixture into a tube of hard glass, No. 3, about 12 centimeters long, and closed at one end. (For the manipulation of tubing, see Appendix, §§ 1-4.) Hold the tube by the open end with the wooden nippers, and heat the mixture gently over the gas lamp. When no further change takes place, allow the tube to cool, break it, and examine the contents.

2. Heat a small piece of lead foil on the lid of a porcelain crucible as long as any change occurs. Warm slowly, as sudden heat will cause the porcelain to break. While this is going on perform the following:

3. Fit to any small flask or bottle a perforated cork (for the manipulation of corks, see Appendix, § 9), to which has been adapted a short piece of glass tubing, No. 7. Slip over the end of this glass tube a short piece of caoutchouc tubing. Suck part of the air out of the flask, and then nip the caoutchouc tube with thumb and finger, so that no air shall re-enter. Immerse the neck of the flask in a basin of water, and release the caoutchouc tube.

4. Take the bottle as prepared for Expt. 3, and into the upper end of the bit of caoutchouc tubing insert the point of a small funnel. Fill the funnel with water; report the result; loosen the cork; report what happens.

EXERCISE 2. — CHEMICAL AND PHYSICAL CHANGES.

5. Burn some magnesium wire, observe carefully the result of the combustion, and describe the product.

6. Mix thoroughly 4 grams of powdered sulphur and 7 grams of fine iron filings. Examine the mixture with a lens; also with a magnet by putting some of the mixture on a slip of paper, and passing the magnet to and fro on the under side of the paper. Now heat the mixture in an ignition tube under the hood. After the action is over, break the tube and examine its contents with lens and magnet. Place a small portion of the contents in a test tube, and pour upon it 1 c. c. of dilute chlorhydric acid (HCl), and note the result. Keep careful notes.

7. Heat 2 grams of red oxide of mercury in a matrass, thrusting into the matrass a glowing splinter of wood from time to time. Observe carefully all that happens.

8. Upon a small piece of "quicklime" put a few drops of water. Note the result.

9. Heat a crystal of copper sulphate *gently* on a crucible cover. When the mass is cool, add a drop of water, holding the cover in the palm of the hand.

10. Pour into a test tube 5 c. c. of a solution of copper sulphate (CuSO_4); add 4 or 5 drops of ammonia water, and shake; finally add 3 c. c. of ammonia water, and shake. Record all that you observe.

11. Proceed as in Expt. 10, but use 5 c. c. of a solution of acetate of lead and 1 c. c. of a solution of chromate of potassium instead of sulphate of copper and ammonia water.

12. Warm 5 c. c. of a solution of potassium permanganate in a test tube until you can just bear your hand on it; then add three or four drops of dilute sulphuric acid, and finally a few crystals of oxalic acid.

EXERCISE 3. — MANIPULATION OF GLASS.

13. Bend glass tubing into the forms shown in the model. Make as many pieces of each form as are designated. These pieces of tubing should be saved, as they are all to be used in subsequent experiments.

Show your work to an instructor and have it approved, before doing other work. For instruction in glass working, read Appendix, pp. 3 to 5 inclusive.

EXERCISE 4. — HYDROGEN.

14. Make a small cylinder of wire gauze, by rolling a piece of fine gauze, about 6 c. m. square, around a thick piece of No. 6 glass tubing. Twist fine wire around the cylinder in order to preserve its form; then slip the cylinder off the glass and close one end of it by pressure with a stout pair of pincers. Within this cylinder of wire gauze place a piece of metallic sodium as large as a pea, and then close the upper end of the cylinder by pressure with the pincers, as before.

Attach the wire gauze cage firmly to the end of a piece of stout iron wire, and thrust it quickly into the water pan, so that the cage will come directly under the mouth of a small bottle of about 100 c. c. capacity, which has been previously filled with water, and is held inverted in the pan. Gas will collect and is to be tested with a lighted match.

15. Experiment indicated under § 23.* Take an iron "gas pipe" and fill the middle portion with bright iron turnings. Rest the tube on the furnace lamp (without the iron trough) and connect one end with the water pan, as in the last experiment. The other end is connected, by means of delivery tubing and caoutchouc connectors, with a round-bottomed flask, half full of water, and supported on a ring of the iron stand. The flask should rest on a piece of wire gauze, and the bottom of the flask should be about 4 or 5 c. m. above the top of the Bunsen lamp. Light the furnace lamp, and wait until the iron pipe has become red hot; then heat the water in the flask until it boils slowly. As the steam passes over the hot iron turnings it will be decomposed; a gas will pass off through the delivery tube, and is to be collected in bottles at the water pan as soon as the air originally contained in the tubes and flask has all been expelled. Test this gas with a lighted match.

16. Prepare a hydrogen generator similar to the pattern shown. Within the bottle put 15 or 20 grams of granulated zinc, or small scraps of the sheet metal, and as much water as will fill about one quarter of the bottle. Replace the cork in the bottle, taking care to press it in tightly, and satisfy yourself that the apparatus is perfectly tight before proceeding further. Record your method of testing. Then, and not till

* Eliot and Storer's Manual.

then, gradually pour in common muriatic acid through the thistle tube. The thistle tube must reach nearly to the bottom of the bottle, so that its point may dip beneath the water; and the muriatic acid must be added by small successive portions—not more than a large thimbleful at a time. On the addition of the first portions of the acid, chemical action will ensue, and after all the air has been expelled from the bottle, the hydrogen may be collected over the water pan in inverted bottles filled with water. The moment at which the hydrogen ceases to be contaminated with air can be determined by collecting small portions of the escaping gas in wide-mouthed bottles of about 50 c. c. capacity, and testing its quality by means of a lighted match. In doing this the small bottle filled with gas must not be turned over, but should be carefully lifted from the water without changing its vertical position, and the lighted match should then be applied to the mouth of the bottle. If the hydrogen be pure, it will burn tranquilly at the mouth of, and within, the bottle; but, in case the gas is still mixed with much air, a sharp explosion will occur at the moment when the match is touched to it. In experimenting with hydrogen, no light should ever be brought into contact with the contents of the bottle in which it is generated, or with any large quantity of the gas, until the purity of the sample, or rather its non-explosive character, has been demonstrated by applying to a very small volume of the gas the test above described.

17. Carefully lift from the water pan a bottle of 200 or 300 c. c. capacity, completely full of hydrogen; slowly carry the bottle, the mouth of which is, of course, held downward, to a burning candle or splinter of wood, and depress the bottle over this flame. After observing what happens, withdraw the candle slowly.

18. Take a glass tube 3 or 4 c. m. in diameter, and close one end with a plug of plaster of Paris 1 or 2 c. m. thick. Set the tube aside to dry until the next exercise.

EXERCISE 5. — HYDROGEN.

19. Fill the plugged tube (prepared at the last exercise) with hydrogen by upward displacement, taking care not to wet the plug. Set the tube upright in a bottle of water, and allow it to remain until the close of the exercise, noting its condition from time to time.

20. Over a jet of burning hydrogen hold a dry cold bottle.

21. Introduce two volumes of hydrogen and five volumes of air into a strong bottle, such as is used for soda water. Close the mouth of the bottle with a cork, and shake violently, in order that the gases shall be mixed. A small quantity of water should be left in the bottle to act as a stirrer. Grasp the bottle firmly in one hand, remove the cork with the other, and apply the open mouth of the bottle to a lighted candle.

22. Light a small jet of hydrogen. Place over the jet a piece of ignition tube 30 or 40 c. m. long, and move the jet up and down within the tube until a place is found where a musical tone is given.

23. Place 5 grams of copper oxide in a piece of ignition tubing about 35 c. m. long; place the tube upon a furnace lamp, heat, and pass a stream of dry hydrogen through the tube until no more water is given off. Allow to cool and weigh the contents of the tube

EXERCISE 6. — CHLORINE.

[All experiments in this exercise must be performed under the hood.]

24. Place in a test tube 1 gram of potassium bichromate ($K_2Cr_2O_7$). Pour upon it 10 c. c. of hydrochloric acid (HCl), and warm it. Note the result.

25. In a flask of about 500 c. c. capacity, arranged as in the model on the desk, place 8 or 10 grams of coarsely powdered manganese binoxide; pour upon it 50 or 60 c. c. of common muriatic acid, and gently heat the mixture. Chlorine will soon be disengaged, and may be recognized by its peculiar color. Fill several bottles of at least half a liter capacity with the dry gas, by displacement.

It is necessary, for the success of this experiment, that the gas be thoroughly dried; this is effected by heating the flask containing the manganese binoxide and chlorhydric acid *gently*, and passing the chlorine through a tube filled with chloride of calcium or other substances which will absorb moisture. Gradually sift a gram or two of very finely powdered metallic antimony into one bottle.

Drop a piece of Dutch metal foil into a jar of chlorine.

Introduce a thin slice of phosphorus into a bottle of dry chlorine.

Into another bottle of chlorine thrust a burning taper or a bit of flaming wood or paper, or better a burning candle.

26. Generate hydrogen as in Expt. 16; when the hydrogen is found by testing, to be pure, connect with the generator a tube drawn to a fine point. Light this jet of hydrogen and immerse it in a bottle of chlorine. Apply a piece of litmus paper to the sides of the bottle.

Repeat, using a jet of illuminating gas.

27. In a soda water bottle mix equal volumes of chlorine and hydrogen; then remove the cork and hold the mouth of the bottle in the flame of a lamp.

28. Suspend a piece of calico in a jar of perfectly dry chlorine. See if there is any change. Now wet the calico; and again suspend it in the chlorine. Explain the result.

EXERCISE 7.—CHLORINE.

29. At the bottom of a large, tall bottle, or other wide-mouthed glass vessel, of the capacity of two or three liters, place a small bottle containing 15 or 20 grams of bleaching powder. Cover the beaker with a glass plate or sheet of pasteboard, provided with a small hole at the center; through this hole in the cover pass a thistle tube down into the bottle of bleaching powder and pour upon it several small successive portions of sulphuric acid diluted with an equal volume of water. Chlorine gas will immediately be set free and is to be ladled out of the jar with a dipper made of a small bottle and poured upon a solution of indigo to show its bleaching power.

30. Soak a bit of printed calico in a half liter of water, into which 10 or 15 grams of bleaching powder have been stirred. After a short time transfer the cloth to another bottle filled with very dilute chlorhydric or sulphuric acid. Finally, wash the cloth thoroughly in water.

31. Pour into a test tube a quantity of chlorine water; drop into it a small quantity of a solution of indigo, and stir the mixture with a glass rod. Repeat with solutions of aniline red, aniline violet, cochineal, log-wood, and potassium permanganate.

After you have performed all the above experiments, perform any which you may have omitted in the previous exercise.

EXERCISE 8. — BROMINE AND IODINE.

32. From an assistant receive into a flask or bottle of 1 or 2 liters capacity 3 or 4 drops of bromine. Cover the bottle loosely and leave it standing; immerse a piece of moist litmus paper in the gas.

33. Warm gently a few crystals of KBr with 0.2 gram MnO_2 and 1 c. c. H_2SO_4 in a test tube and observe the vapor. (Under the hood.)

34. Take 5 c. c. of bromine water in a tube and warm under the hood. Observe what happens. Test the remaining liquid with litmus paper.

35. Receive a few drops of dry bromine in an ignition tube; throw upon it a little powdered antimony.

36. Hold a dry test tube in the gas lamp by means of the wooden nippers and warm it along its entire length, in so far as this is practicable. Drop into the hot tube a small fragment of iodine.

37. Prepare a quantity of thin starch paste by boiling 30 c. c. of water in a porcelain dish and stirring into it 0.5 gram of starch which has previously been reduced to the consistency of cream by rubbing it in a mortar with a few drops of water. Note the change in the starch.

38. Pour 3 or 4 drops of the paste into 10 c. c. of water in a test tube, and shake the mixture so that the paste may be equably diffused through the water; then add a drop of an aqueous solution of iodine. Heat the solution gently until the color disappears, and allow it to cool again. This action affords a delicate test for iodine when not in combination.

39. Dip a strip of white paper in the starch paste and suspend it, while still moist, in a large bottle, into the bottom of which two or three crystals of iodine have been thrown. What does the experiment show?

40. To a portion of the starch paste made in Expt. 37 add a few drops of potassium iodide solution. Into the paste thus prepared dip strips of filter paper. This is "iodo-starch paper."

41. Repeat Expt. 38, using bromine water instead of the aqueous solution of iodine.

42. Put 1 gram of MnO_2 in a test tube, pour upon it 1 c. c. of HCl , and warm gently. Now hold a piece of iodo-starch paper over the tube and notice the result. Explain the action. This affords a test for chlorine.

43. Take a very dilute solution of potassium iodide, add a little carbon bisulphide and a few drops of chlorine water; shake up and

then allow to stand for a few moments. Repeat, using potassium bromide instead of the iodide. Chloroform may be used instead of carbon bisulphide. Their action is merely mechanical. This shows how we may recognize bromine and iodine in compounds.

EXERCISE 9. — IODINE AND FLUORINE.

44. Place 0.25 gram of finely powdered iodine in a porcelain capsule, pour upon it enough concentrated ammonia water to somewhat more than cover the iodine, and allow the mixture to stand during 15 or 20 minutes with occasional stirring. Collect in several small filters (Appendix, § 15) the insoluble dark brown powder which will be found at the bottom of the liquid. Wash well with cold water and then remove the filters, together with their contents, from the funnels; pin them upon bits of board and allow them to dry spontaneously. The powder is the nitrogen iodide. As soon as it has become thoroughly dry, it will explode upon being rubbed, even with a feather, or jarred, as by the shutting of a door or by a blow upon the wall or table.

45. [Under the hood.] Warm a slip of glass and rub it with beeswax so that it shall be everywhere covered with a thin, uniform layer of the wax. With a needle, or other pointed instrument, write a name, or trace any outline through the wax, so as to expose a portion of the glass. Lay the etching, face downward, upon a bowl or trough of sheet-lead, in which has been placed a teaspoonful of powdered fluor-spar and enough strong sulphuric acid to convert it into a thin paste.

Cover the glass and the top of the dish with a sheet of paper, and then heat the leaden vessel for a few moments very gently, taking care not to melt the wax; then set the dish aside in a warm place and leave it at rest until the end of the exercise. Finally, melt the wax and wipe it off the glass with a towel or bit of paper.

46. Into a perfectly dry matrass put a small quantity of a mixture of equal parts of quartz and powdered fluor-spar. Moisten with a drop of strong sulphuric acid and heat in the flame of the lamp. Hold a drop of water in a loop of platinum wire at the mouth of the tube. Show result and ask for explanation. This shows how we may recognize fluorides. The experiment will be performed in a subsequent exercise, and will then be fully explained.

EXERCISE 10. — CHLORHYDRIC ACID.

47. Put 30 grams of dry, coarse salt into a flask of a liter capacity. Arrange the apparatus as shown in the pattern. When you are sure that the apparatus is all right, pour 50 grams of strong sulphuric acid upon the salt, and immediately cork the flask, place it upon a sand bath on the iron stand and connect the delivery tube with the Woulfe bottles.

Be sure that the tube from the flask does not dip into the water of the first bottle. The contents of the flask must be *very gradually* and moderately heated, else a violent frothing is liable to occur, which would spoil the experiment. In your notes describe the apparatus; and show the use of safety tubes.

48. Perform Expt. 47, and while the gas is coming off disconnect the flask for a moment and bring an open bottle of NH_4HO into the neighborhood. Also, while the experiment is going on, put into separate test tubes small fragments of CaCl_2 and NH_4Cl , cover with strong H_2SO_4 , warm *gently* and test for evolved gas by smell and by moistened red and blue litmus paper.

49. Pour a few c. c. of the contents of the Woulfe bottle nearest the flask into a test tube, add a strip of zinc and note the result.

50. To 5 c. c. of the contents of the first Woulfe bottle add 5 drops of a solution of silver nitrate (AgNO_3) and shake violently, and note the result. Save the contents of the test tube. Put it into the jar provided marked "silver residues."

51. Into each of two test tubes put a small bit of gold leaf. Into one test tube put one or two c. c. of strong nitric acid; into the other put an equal quantity of strong chlorhydric acid. Warm gently. Finally mix the contents of the two tubes. The mixture of nitric and chlorhydric acid is known as *aqua regia*.

52. Place 10 c. c. of a solution of acetate of lead in a test tube and add dilute HCl until no further precipitation takes place. Let the precipitate settle, then pour off the liquid; add ten c. c. of water to the precipitate and boil for a minute or two, and then filter quickly but carefully, receiving the filtrate in a clean test tube kept warm by being immersed in a beaker of hot water. When the liquid has filtered through, remove test tube from the hot water and allow to cool.

EXERCISE II. — OXYGEN AND OZONE.

53. In a clean bottle of 1 or 2 liters capacity place a piece of phosphorus 2 or 3 c. m. long, the surface of which has been scraped clean (under water) with a knife; pour water into the bottle until the phosphorus is half covered; cover the bottle with a plate of glass and set it aside in a place where the temperature is 20° or 30° . Observe the appearance and odor of the contents of the bottle after 15 or 20 minutes. Suspend in the bottle a strip of iodo-starch paper, and also a strip of red litmus paper which is saturated with potassium iodide solution. Explain the results. During this experiment perform those following.

54. Mix intimately 5 grams of potassium chlorate with 5 grams of "black oxide of manganese," which has been previously well dried. Place the mixture in a tube of hard glass, No. 1, 12 or 15 c. m. in length. Attach to this ignition tube, by means of a perforated cork or caoutchouc stopper, a delivery tube of glass, No. 7, as in the model. Heat the mixture in the ignition tube, and collect the gas which will be given off in bottles or jars of the capacity of about 250 c. c. The first 100 c. c. or so of gas should be rejected, since it will be contaminated with the air originally contained in the apparatus. Collect at least *five* bottles and one test tube full of the gas for subsequent experiments.

In performing this experiment the following precautions should be observed: 1. Both the potassium chlorate and the manganese binoxide should be perfectly dry and pure; that is, free from moisture, dust or particles of organic matter. They should not be ground together under any circumstances.—DANGER! 2. As soon as the gas begins to be delivered, the heat beneath the ignition tube should be diminished, if need be, and so regulated that the evolution of gas shall be tranquil and uniform. 3. The uppermost portions of the mixture should be heated before the lower. 4. The ignition tube should never be filled to more than one third its total capacity, lest solid matter be projected into the delivery tube, and the outlet for the gas be thus stopped. 5. The ignition tube should always be inclined and never placed upright in the flame.

Place in a deflagrating spoon a bit of sulphur as large as a pea. Light the sulphur and thrust it into a bottle of oxygen.

Burn a bit of phosphorus in oxygen in a similar manner, but with much greater care.

Place a piece of charcoal—that of bark is best—in a deflagrating spoon. Kindle the charcoal by holding it in the flame of a lamp and then introduce it into a bottle of oxygen.

Make into a spiral coil some fine iron wire—or, better, a watchspring, which has been rendered flexible by igniting it and allowing it to cool slowly—and to the end attach a bit of tinder or the tipped end of a match. Light the kindling material and plunge the spiral into a jar of oxygen in the bottom of which an inch or so of water has been left.

55. Support a rather wide tube of thin glass—the neck of a broken retort, for example—in a vertical position and connect the upper opening with a gas generator from which hydrogen is being evolved. Allow the gas to flow until the tube is filled; then apply a lighted match to the mouth of the tube, and regulate the flow of gas so that the latter may continue to burn slowly at the lower edge of the tube. Meanwhile, prepare some oxygen as in Expt. 54, connecting the ignition tube with a piece of narrow glass tubing, drawn out to a fine point; and, while the oxygen is flowing through this tube, pass it up into the larger tube filled with hydrogen. This experiment should be performed by two students together as will be directed.

56. Dissolve a bit of potassium hydrate (KOH) in a few c. c. of water, add a teaspoonful of pyrogalllic acid, and pour the solution into the test tube containing the O, taking care that the O shall not escape. Place the thumb over the end of the tube and shake. Notice the result. Place the mouth of the tube under water and remove the thumb, and observe what happens.

57. Heat some black oxide of manganese in a small matrass in the blast lamp, and see whether oxygen is evolved.

EXERCISE 12. — SULPHUR.

58. Place in a test tube, of about 30 c. c. capacity, 15 to 20 grams of coarsely powdered sulphur; melt the sulphur slowly over the gas lamp, and continue to heat it until it begins to boil, noting, meanwhile, the changes which the sulphur undergoes. Finally, pour the hot sulphur, in a fine stream, into a large dish full of cold water, and examine its appearance.

59. In a small beaker glass, or Hessian crucible, slowly heat 50 to 60 grams of sulphur until it has entirely melted. Remove the vessel from the lamp and allow it to cool slowly until about a quarter part of

the sulphur has solidified; then pour off into a basin of water that portion of the sulphur which is still liquid, breaking through, for this purpose, the crust at the top of the liquid, if any such have formed. Show the result.

60. In a test tube, melt enough sulphur to fill one quarter of the tube; place the tube in such a position that its contents may cool slowly and quietly, and then watch the formation of crystals as they shoot out from the comparatively cold walls of the tube towards the center of the liquid.

61. Place 1 gram of sulphur in a dry test tube and pour upon it 5 c. c. of carbon bisulphide, cork tightly, and shake for a few moments. [Carbon bisulphide is volatile and VERY INFLAMMABLE. HAVE NO LIGHTS NEAR BY.] Pour a little of the clear solution upon a watch glass and allow the carbon bisulphide to evaporate under the hood. Examine the residue obtained.

62. Place a few bits of sulphur in a perfectly dry test tube and heat gradually until the sulphur boils. Notice the sublimate.

63. Mix intimately 4 grams of flowers of sulphur and 7 grams of the finest iron filings. Place the mixture in an ignition tube 10 to 12 c. m. long, and heat the lower end of the tube gently over the gas lamp under the hood. When chemical action once begins, take the tube out of the flame. Don't be surprised if the tube breaks.

64. Heat a gram or so of sulphur in an ignition tube until it boils, and allow a few bits of copper filings to fall into the melted sulphur.

65. Heat some powdered pyrite in a matrass.

EXERCISE 13. — HYDROGEN SULPHIDE.

66. Put 10 or 12 grams of iron sulphide into a generator bottle. Arrange the delivery tube so that it shall dip beneath the surface of water contained in another (smaller) bottle. Through the thistle tube, pour into the gas bottle water enough to seal the lower extremity of this tube; then add, through the thistle tube as before, 10 c. c. of strong chlorhydric acid, and observe. When the disengagement of gas slackens, a new portion of chlorhydric acid may be added through the thistle tube, and this process continued until the water in the absorption bottle smells strongly of the gas.

[The experiment must be performed under the hood.]

67. To the delivery tube of the gas bottle employed in generating hydrogen sulphide, attach a drying tube containing fragments of calcium chloride, and with the tube connect a piece of No. 6 glass tubing drawn out to a fine point. When the apparatus is full of the gas, apply a match to the end of the tube. The gas will take fire. Hold a dry bottle over the flame, and test the deposited moisture with litmus paper.

68. Put a drop of sulphureted hydrogen water on a bright piece of lead, copper, or silver.

69. Dissolve a small crystal of lead nitrate in a test tube half full of water, and to this solution add a few drops of the sulphureted hydrogen water.

70. Take 20 c.c. of a solution of each of the following substances: CuSO_4 , As_2O_3 , SbCl_3 , ZnSO_4 , MnSO_4 , K_2SO_4 . Place each solution in a separate test tube and saturate each with H_2S . Have solutions boiling hot. Provide a short piece of delivery tube for each test tube, so that there will be no danger of mixing the solutions. Observe what happens; write the reactions. Then add 1 c.c. strong HCl to each tube, shake, and observe the effect.

71. Moisten a piece of filter paper with acetate of lead solution and hold it near the delivery tube of an H_2S generator. This is the test for H_2S .

72. Pass H_2S through 10 c.c. of HNO_3 (1 : 1).

73. Prepare apparatus exactly as in Expt. 67, except make the tube which ends in the jet straight and about 20 c.m. long. Heat this tube gently with the Bunsen burner near the center while a stream of H_2S is passing through it, and observe the tube carefully.

EXERCISE 14. — NITROGEN.

74. Into a small porcelain capsule, supported on a piece of stout iron wire as will be shown, put about a cubic centimeter of phosphorus, and set it on fire. Invert over the capsule a wide-mouthed bottle, of the capacity of a liter or more, and hold this bottle so that its mouth shall dip beneath the surface of the water. The dense white cloud which fills the bottle at first is a compound of phosphorus and oxygen, which is soluble in water. It will, therefore, soon be absorbed by the water in the pan, and will disappear. Remove the wire with the capsule, which may be readily done by tipping the bottle to one side, taking care that the mouth does not come out of the water, and slip a glass plate under

the mouth of the bottle; invert the bottle so that it stands upright, and thrust a burning splinter of wood or a lighted candle into the gas.

75. Take a piece of ignition tubing about 35 centimeters long, and introduce into it enough copper turnings to fill loosely about 15 centimeters of its length. Support the tube upon one of the "furnace lamps," resting the tube in an iron trough. Into one end of the tube fit a cork through which passes a bent delivery tube leading to the water pan; connect the other end of the tube with a "gasholder bottle" similar to that shown in the model. Light the furnace lamp and heat the tube gently until the iron trough becomes red hot. Then pass a *slow* current of air over the heated copper, which is accomplished by pouring water into the gasholder bottle. Collect in bottles the gas which is delivered at the water pan, and test it with lighted splinters.

76. Put 3 c. c. of ammoniacal cuprous chloride into a test tube, close the end with the thumb, and shake vigorously. Invert the tube in the water pan and remove the thumb. Note the result. Test the gas remaining in the tube with a lighted splinter.

77. Determine accurately the contents of a narrow test tube in cubic centimeters by filling it with water from a burette, and pour into the test tube exactly 5 c. c. of potassium pyrogallate solution. Shake carefully for several minutes, keeping the tube tightly closed with the thumb. Now open under water as in Expt. 56, and allow the water to rise. Then place the thumb underneath the tube and raise it from the pan and invert it, still holding the thumb over the end. Determine the volume of the unabsorbed gas by filling the tube with water from a burette. Calculate the percentage of oxygen in the air by volume.

EXERCISE 15. — AMMONIA.

78. Mix thoroughly 25 grams of ammonium chloride with an equal weight of cold freshly slaked lime. Place the mixture in a flask of 500 c. c. capacity, cover with water, connect with a couple of Woulfe bottles, as shown in the pattern, and then heat the flask. Describe the apparatus and explain the action of the various tubes. When the experiment is finished, examine the contents of the Woulfe bottles. Perform the experiment under the hood if possible.

79. While Expt. 78 is going on, disconnect the flask for a moment and bring an open bottle of strong HCl into the neighborhood. Also, fill a test tube with the gas — by upward displacement — close the mouth

tightly with the thumb, place the tube, mouth downwards, in a vessel of water, and then remove the thumb.

80. Mix together equal quantities of NH_4NO_3 and CaH_2O_2 and heat gently in a test tube. Test for evolved gas by smell and by moistened red litmus paper. Also put a little solid NH_4Cl and NH_4NO_3 into separate test tubes, cover with a solution of NaHO in each case, and warm gently, testing as in previous instance.

82. Heat in an ignition tube 7 grams of fine iron filings and 0.5 gram powdered KOH . Collect the gas in test tubes over the water pan, and test it with lighted splinter. Perform the same experiment with 7 grams of coarse iron filings and 0.5 gram KNO_3 . Now heat a mixture of 14 grams coarse iron filings, 0.5 gram KOH and 0.5 gram KNO_3 in a large test tube, and test the gas evolved as in Expt. 79.

EXERCISE 16. — PHOSPHORUS.

[Extreme care must be taken in all experiments with phosphorus.]

83. Put a piece of phosphorus as big as a grain of wheat upon a piece of filter paper and sprinkle over it some lamp-black or powdered bone-black. Allow it to stand for a while.

84. In a narrow glass tube, No. 6, about 30 c. m. long and closed at one end, place a quantity of red phosphorus as large as a small pea; heat the phosphorus gently over the gas lamp and note the character of the sublimate which forms. When the tube is perfectly cold, cut it off just below the sublimate and scratch the coating with an iron wire.

85. In order to observe the comparative difficulty of inflaming red phosphorus, lay an inverted cover of a porcelain crucible upon an iron triangle upon the lamp stand; place upon the cover, at some distance from each other, a small bit of ordinary phosphorus and an equal quantity of red phosphorus; heat the cover *gently and gradually* over the gas lamp. [This experiment should be performed under the hood, if possible.]

86. In a thin-bottomed flask of about 140 c. c. capacity, put 1 gram of phosphorus and 115 c. c. of hydrate of sodium, obtained by dissolving 40 grams of common caustic soda in 110 c. c. of water. Pour two or

three drops of ether upon the liquid in the flask, then close the flask with a cork carrying a long delivery tube of glass, No. 5. Place the flask over the gas lamp upon a piece of wire gauze, and immerse the end of the delivery tube in the water pan; then gently heat the flask. The ether is added in order that the last traces of air may be expelled from the flask by the heavy vapor into which this highly volatile liquid is converted as soon as it is warmed. As soon as bubbles of gas begin to be expelled, the heat should be moderated or the lamp wholly removed. Perform the experiment under the hood, if possible.

EXERCISE 17. — SULPHUROUS ANHYDRIDE.

87. Light a piece of sulphur in a deflagrating spoon and suspend the latter in a two liter bottle full of air. Observe the odor. Immerse a lighted taper in the gas obtained.

88. Place a pinch of copper filings in a test tube, cover with concentrated H_2SO_4 , and warm GENTLY. Observe the odor of SO_2 . Endeavor to bleach a rose or other flower, previously moistened, by holding it in the fumes.

89. Repeat Expt. 88, but use charcoal in the place of the copper.

90. Charge a bottle, of the capacity of a liter or more, with sulphurous acid by burning in it a bit of sulphur. Fasten a shaving, or, better, a tuft of gun-cotton, upon a glass rod or tube bent at one end in the form of a hook; wet the shaving in concentrated nitric acid, and hang it in the bottle of sulphurous acid. Interpret what you observe.

Pour a little BaCl_2 solution into the bottle before beginning the experiment, and notice its condition at the end after shaking the bottle. Write the reaction.

91. Arrange a generator flask as shown in the model. Place in the bottom 15 grams of charcoal and add 50 c. c. of concentrated H_2SO_4 . Warm gently upon a sand bath, and SO_2 will be evolved. Place 5 grams of PbO_2 in a piece of ignition tube, and pass a slow current of SO_2 through the tube, and at the same time heat the tube containing the PbO_2 moderately upon a furnace lamp. *Perform this experiment under the hood.*

EXERCISE 18. — SULPHURIC ACID.

92. Place in a pint bowl 30 c.c. of water; pour gradually into the water 120 grams of concentrated sulphuric acid, stirring the mixture with a narrow test tube containing a few c. c. of water. So much heat will be evolved during the union of the water and the acid that the water in the test tube may actually boil. Save the dilute acid.

93. Pour 30 grams of strong H_2SO_4 upon 120 grams of snow, and observe the temperature of the mixture with the thermometer.

94. Repeat Expt. 93, but use 120 grams of H_2SO_4 and 30 grams of snow. Save the acid.

95. Into a test glass pour a tablespoonful of sulphuric acid, and immerse in it a splinter of wood.

96. Place 5 grams of sugar in an evaporating dish and pour 10 c. c. of strong sulphuric acid upon it.

97. Place 27 c. c. of water in a beaker, and add slowly 73 c. c. of concentrated H_2SO_4 . When cool, transfer the mixture to a graduate, and note the volume. Save the acid.

98. Pour a few drops of H_2SO_4 into a test tube and dilute with 20 c. c. of water. Add a little BaCl_2 solution, and observe the effect. This is the test for H_2SO_4 . (See Expt. 90.)

99. Dissolve a crystal of PbN_2O_6 in water in a test tube, and add a few drops of dilute H_2SO_4 .

100. Boil 0.2 gram flowers of sulphur with 1 c. c. concentrated HNO_3 , dilute with 20 c. c. of water, and add a few drops of BaCl_2 solution.

EXERCISE 19. — PHOSPHORIC ACID.

101. Dry thoroughly a large porcelain plate, a small porcelain capsule, and a wide-mouthed bottle of two liters capacity. Put in the capsule a bit of dry phosphorus, of the weight of about half a gram, and place the capsule on the plate; light the phosphorus, and cover it at once with the inverted bottle. Drop a little of the white powder obtained into water. Test the solution with litmus paper.

102. Touch a piece of blue litmus paper to a piece of moist phosphorus.

103. To 10 c. c. of Na_2HPO_4 solution add BaCl_2 in excess. Filter the mixture, and test the solubility of the precipitate in ammonia water and in dilute chlorhydric acid.

104. (I) To 2 c. c. of Na_2HPO_4 add 10 c. c. of water, and 2 c. c. of NH_4Cl , and 1 c. c. of ammonia water. Now add slowly 3 c. c. of a mixed solution containing MgSO_4 and NH_4Cl . Observe carefully under the microscope the character of the precipitate formed. Filter and test as in 103.

105. (II) To 5 c. c. of Na_2HPO_4 , add AgNO_3 solution in slight excess. Note the color of the precipitate. Filter and test its solubility in ammonia water, dilute nitric acid and dilute chlorhydric acid. Save the silver residues.

106. (III) To a few c. c. of Na_2HPO_4 add an equal volume of a solution of molybdate of ammonium in nitric acid. [The last three experiments are the usual tests for phosphoric acid and phosphates.]

107. To a few c. c. of Na_2HPO_4 add a few c. c. of ferric chloride solution. Now add ten c. c. of $\text{NaC}_2\text{H}_3\text{O}_2$ solution, and observe the effect. Test the solubility of the precipitate in HCl and in $\text{HO}(\text{C}_2\text{H}_3\text{O})$.

108. Weigh a small porcelain dish very carefully. Next weigh out 15 grams of crystallized sodic phosphate and place it in the dish. Heat carefully over the burner until the substance is reduced to a white mass, and then weigh. Heat again for a few moments, and repeat the weighing until the weight of the dish containing the substance shows no further diminution. Calculate the per cent of water that has been driven out. Fuse a little of the dry substance on platinum foil and then dissolve it in water and add AgNO_3 , and compare the precipitate with that obtained in Expt. 105. Ask for explanation. Save the silver residues.

EXERCISE 20. — NITRIC ACID.

109. Into a tubulated glass-stoppered retort of 250 c. c. capacity put 40 grams of powdered potassium nitrate, or, better, 34 grams of powdered sodium nitrate, if it can be obtained; and through the tubulure pour 50 grams of strong sulphuric acid, which has been weighed out in a bottle previously counterpoised upon the balance with shot or coarse sand. Imbed the bottom of the retort in sand contained in a small iron pan placed over the gas lamp on a ring of the iron stand. Thrust the neck of the retort into a flask of from 500 to 700 c. c. capacity; the retort neck should fit quite loosely in the neck of the flask in order to avoid the possibility of any pressure being created within the retort during the operation. Place the flask in a pan of cold water, and cover it with cloth or

bibulous paper, which must be kept wet during the distillation. Heat the sand bath *moderately* (that the frothing which occurs may not become too violent). When all frothing has ceased, and the mass in the retort is in a state of tranquil fusion, while very little liquid passes over into the receiver, the lamp is to be put out. Allow the retort to cool on the sand bath, and do not attempt to wash it out.

Transfer the nitric acid to a tared beaker and weigh. Put the acid into the bottle prepared for it. Calculate the amount which should be obtained theoretically. Save the acid for further experiments.

110. To 1 c. c. of the nitric acid of Expt. 109 add 10 c. c. of water. Touch a drop of the mixture to the tip of the tongue. Dip a piece of blue litmus paper into the liquid.

111. Dissolve about one gram of caustic potash in 20 c. c. of water. Rub a little of the solution between the fingers. To one c. c. of this solution add 20 c. c. of water, and cautiously taste of it. Immerse a piece of red litmus paper in it.

112. Into a flask of about 500 c. c. capacity put 50 c. c. of strong nitric acid and 5 grams of starch. Warm the flask gently, and as soon as the mixture begins to turn reddish brown remove the lamp. The experiment must be performed under the hood. When the flask is cool, fill it with water and empty it into the sink under the hood.

113. Treat a piece of lead foil in a test tube with HNO_3 , strong and dilute, and observe the result.

114. To 5 c. c. of indigo solution in a test tube, add 1 c. c. of HNO_3 and warm.

EXERCISE 21. — NITRIC ACID.

[Hereafter write the equations when possible.]

115. To 5 c. c. of nitric acid, diluted with twice its bulk of water, add cautiously a rather dilute solution of caustic potash (potassium hydrate, KHO) until the mixture turns litmus paper neither red nor blue. Evaporate the solution in a porcelain dish, taking care that the liquid does not actually boil, until a drop taken out on the end of a glass rod becomes nearly solid on cooling. Then remove the lamp, and allow the dish to become cold. Show the result, and learn the significance of the experiment.

116. Take 5 c. c. of the nitric acid, diluted as in Expt. 115, then add 10 grams of PbO . Warm the mixture and filter the liquid, while hot, into

another evaporating dish. Evaporate the solution carefully, but take away the lamp when four fifths of the liquid have disappeared.

117. Fill a perfectly dry ignition tube about one third full of lead nitrate which has been finely powdered and *thoroughly dried*. Connect the ignition tube with a dry bottle and finally with the water pan, as shown in the model. The small bottle must be surrounded by a mixture of ice (or snow) and salt. Heat the ignition tube gently, and when the evolution of gas has once begun, care must be taken that the tube is not suffered to cool, so as to allow the water to suck back from the water pan. Red fumes will fill the delivery tubes, and will condense in the small bottle to a brownish yellow liquid if the experiment is successful. Test the gas which collects at the water pan.

118. Neutralize 1 c.c. of dilute nitric acid with ammonium hydrate, evaporate to dryness, and heat a portion of the residue gently upon platinum foil.

EXERCISE 22. — OXIDES OF NITROGEN.

119. Into a *dry* flask of thin glass of about 300 c.c. capacity, introduce 10 or 15 grams of ammonium nitrate. From the flask carry a delivery tube, No. 6, to the water pan; but interrupt the tube at some convenient point to interpose, by means of a cork or caoutchouc stopper with two holes, a small bottle, which can be kept cool with water. Heat the flask moderately and cautiously. When gas begins to escape from the melted mass, the heat must be so controlled that the evolution of the gas shall not be tumultuous. Collect the gas in bottles of 300 to 400 c.c. capacity. When two bottles of gas have been filled, the delivery tube may be withdrawn from the water and the lamp extinguished.

Insert a glowing splinter of wood into a bottle of the gas.

Burn a bit of sulphur in nitrous oxide.

Test the liquid collected in the interposed bottle.

120. Heat a bit of ammonium nitrate gently on platinum foil, and note the result.

121. Place 15 or 20 grams of copper turnings or filings in a bottle arranged precisely as for generating hydrogen, and pour about 25 c.c. of dilute nitric acid made by adding to the common strong acid its own bulk of water. Collect three bottles, of 300 to 400 c.c. capacity, of the evolved gas, adding acid from time to time as may be necessary. Save the blue solution (copper nitrate) which remains in the generator.

122. Dip a lighted candle into a bottle of the gas. Into the same bottle thrust a glowing splinter.

123. Lift a bottle of the gas from the water so that air may enter the bottle and the gas may escape into the air. Bring into contact with the fumes a piece of moistened litmus paper.

124. Thoroughly ignite a bit of sulphur in a deflagrating spoon, and introduce it into a bottle of the gas. Into the same bottle thrust a piece of phosphorus as big as a pea, burning *actively*.

125. Fill a test tube with a concentrated acid solution of ferrous sulphate. Invert the tube in a water pan over a delivery tube, from which NO is issuing. When half of the liquid has been driven from the tube, place your thumb over the bottom and remove the tube from the pan and shake. Boil the contents of the tube. Notice all the changes.

EXERCISE 23. — CARBON.

126. Put into an ignition tube 12 or 15 c. m. in length, enough bituminous coal, in coarse powder, to fill one third of the tube. Fit to this ignition tube a large delivery tube, and support the apparatus upon the iron stand, having the ignition tube nearly horizontal. Heat the tube and collect in bottles the gas which will be evolved. Test the gas with a lighted match.

As soon as gas ceases to be given off from the coal, take the end of the delivery tube out of the water, and when the ignition tube has become cold, break it and examine the coke which it contains.

127. Repeat the previous experiment, using wood shavings or sawdust instead of coal. Collect and test the gas evolved. After the flow of gas has ceased, remove the end of the delivery tube from the water, plug it so that no air can enter the ignition tube, and lay the apparatus aside until it has become cold. Finally remove the cork from the ignition tube and take out the charcoal which is contained in it. Heat a portion of this charcoal upon platinum foil and note the manner in which it burns.

128. Over a burning candle invert a wide-mouthed bottle of the capacity of a liter or more, one edge of the mouth of the bottle being propped up on a small block of wood, so that some air may enter the bottle. Explain the formation of lamp-black. Also press down upon the flame of an oil lamp or candle an iron spoon or a porcelain plate in

such manner that the flame shall be almost, but not quite, extinguished. Explain the results.

129. Mix two and a half grams of copper oxide with a quarter of a gram of powdered charcoal; place a portion of the mixture in an ignition tube, and heat it strongly in the gas lamp. Test the gas evolved, and examine what is left in the tube.

130. Take from the fire a piece of charcoal which has been heated to full redness for some time; thrust it under water so that it may be suddenly cooled, and compare its behavior with that of a piece of common charcoal. Also, attach a lead sinker to a piece of common charcoal and sink it in a beaker of warm water.

131. Put a small quantity of powdered charcoal into a bottle containing hydrogen sulphide, close the bottle tightly with the palm of the hand and shake for a few moments. Observe how much the odor is affected; observe other attendant phenomena.

132. Provide four small bottles of the capacity of 100 or 200 c. c., and place in each of them a tablespoonful of bone-black; into the first bottle pour a quantity of the blue compound of iodine and starch obtained in Expt. 39; into the second, a decoction of cochineal; into the third, a dilute solution of soluble indigo blue; into the fourth, a solution of blue litmus, of logwood, or indeed of almost any other vegetable coloring matter; enough of the solution being taken in each instance to nearly fill the bottle. Cork the bottles and shake them violently, then pour the contents of each upon a filter and show the several filtrates.

133. Mix 4 grams of potassium nitrate with 2 grams of powdered charcoal. Place the mixture upon an iron plate and touch it with a lighted stick. Perform this experiment under the hood.

EXERCISE 24. — CARBONIC ACID.

134. Place a live coal (charcoal) upon a deflagrating spoon and thrust it into a bottle full of air, or, better, oxygen gas; when the coal has ceased to glow, pour into the bottle some lime water — a solution of common slaked lime in water — and shake the bottle.

135. Mix 11 grams of red oxide of mercury with 0.33 gram of charcoal; heat the mixture in an ignition tube, and collect over water the gas which is evolved. Test the product with lime water, as in Expt. 134. Examine what is left in the tube.

136. In a gas bottle of 500 or 600 c. c. capacity, arranged precisely as for generating hydrogen, place 10 or 12 grams of carbonate of calcium (marble or limestone). Cover the end of the thistle tube with water and then add strong chlorhydric acid by small portions, in such quantity as shall insure a continuous and equable evolution of gas. Collect several bottles of the gas over water, then replace the anterior portion of the delivery tube with a straight tube and collect one or two bottles of the gas by downward displacement.

137. Thrust into a bottle of the gas, obtained in Expt. 136, a lighted candle, or, better, a large flame of alcohol burning upon a tuft of cotton.

138. From a large bottle full of the gas, pour a quantity of carbonic acid upon the flame of a lamp or candle; that is to say, hold the mouth of the open bottle of carbonic acid obliquely over the candle flame, so that the gas shall fall like water upon it.

139. Take a beaker of about 300 c. c. capacity and balance it, very carefully, upon the pan of a balance. Fill a large dry bottle with CO_2 and pour it into the beaker. Notice the increase of weight.

140. Into a long-necked flask or phial filled with carbonic acid, pour a quantity of water, close the bottle with the finger, and shake it; immerse the mouth of the bottle in water and remove the finger; water will rush into the bottle to supply the place of the gas which has been dissolved. Again place the finger upon the mouth of the bottle, shake the bottle as before, and subsequently open it beneath the surface of the water, and so on.

141. Fill a test tube, by displacement, with CO_2 . Pour in 5 c. c. of a solution of KOH, place your thumb over the end, and shake. Immerse the tube in the water pan and remove your thumb.

142. Dissolve 10 grams of honey or molasses in 100 c. c. of water; fill a large test tube with the mixture and add to it a few drops of bakers' or brewers' yeast; close the open mouth of the test tube with the thumb and invert it in a small saucer or porcelain capsule filled with the diluted syrup. Place the saucer and tube, with their contents, in a warm place, having a temperature of about 20° or 30° , and leave them there during 24 hours. In a short time fermentation sets in, and the sugar of the syrup is gradually converted into alcohol and carbonic acid.

143. Take two small bottles and into each put 15 or 20 c. c. of clear lime water. Through one of the bottles force a stream of ordinary air; through the other force a stream of air from the lungs.

EXERCISE 25. — MARSH GAS, ETC.

144. Mix together 2 grams of crystallized sodium acetate, 4 grams of caustic soda, and 8 grams of slaked lime. Heat the mixture gently upon an iron plate, until all the water of crystallization of the acetate has been expelled and the mass has become dry and friable. Charge an ignition tube 20 c. m. long with the dry powder, heat it above the gas lamp, and collect the gas at the water pan. Marsh gas is evolved from the mixture, at a temperature below redness, and a residue of sodium carbonate is left in the ignition tube. The purpose of the lime is to render the mass porous and infusible, or nearly infusible, so that the tube may be heated equably. Attempt to write equation.

145. Fill a tall bottle of at least one liter capacity with warm water, invert it over the water pan and pass marsh gas into it, until a little more than one third of the water is displaced; cover the bottle with a thick towel, to exclude the light, and then fill the rest of the bottle with chlorine. Cork the bottle tightly, and shake it vigorously, in order to mix the gases together, keeping the bottle always covered with the towel. Finally, open the bottle and apply a light to the mixture. After the action has taken place test the moisture on the sides of the bottle.

146. Mix 8 grams of powdered magnesium carbonate with 16 grams of zinc dust. Place the mixture in an ignition tube, tapping it so as to leave a passage over the top for the gas which will be evolved. Heat strongly on a furnace lamp and collect the gas in the ordinary manner over the water pan. Test the gas.

147. Heat 4 grams of CuO in a piece of ignition tubing over a furnace lamp and pass a stream of CO over the hot oxide. Collect the gas over water and test with a lighted splinter and with lime water.

148. Sprinkle fine iron filings into the flame of an alcohol lamp or into the non-luminous flame of the gas lamp. Also, rub together two pieces of charcoal above a non-luminous flame, in such manner that charcoal powder shall fall into the flame.

149. Press down a piece of white letter paper, for an instant, upon the flame of a candle until it almost touches the wick, then quickly remove the paper before it takes fire and observe how its upper surface is charred.

150. Study the flame of a candle. Thrust the phosphorus end of a match into the flame, and see whether you can withdraw it without lighting the match.

EXERCISE 26. — SILICON AND BORON.

151. To a concentrated solution of water glass contained in a small evaporating dish, add enough strong chlorhydric acid to make the solution acid. There will separate a thick jelly-like mass of silicic acid (H_4SiO_4). Evaporate the contents of the dish to dryness on a water bath and then heat the residue gently over the gas lamp. The mass will contract in bulk and, on adding water, there will remain undissolved a fine white powder of silicic anhydride.

152. Take a very dilute solution of water glass and add dilute chlorhydric acid drop by drop until the liquid has a decidedly acid reaction. Compare the result with that of the first part of the previous experiment.

153. Into a perfectly dry matrass put a small quantity of a mixture of equal quantities of quartz and powdered fluor-spar. Moisten with a drop of strong sulphuric acid and heat in the flame of a lamp. Hold a drop of water in a loop of platinum wire at the mouth of the tube. Show result and see blackboard for reactions. This shows how to recognize silicon in its compounds.

154. Dissolve 4 grams of powdered borax in 10 grams of boiling water, in a beaker glass or porcelain capsule of 30 or 40 c. c. capacity, and add to the solution 5 c. c. of concentrated chlorhydric acid. Allow the solution to cool.

155. Dissolve a little crystallized boracic acid in a teaspoonful of alcohol in a small porcelain capsule. Set fire to the alcohol and stir the burning solution with a rod or agitate it by jarring the dish. Repeat, using borax instead of boracic acid. Now put 3 c. c. of H_2SO_4 upon a small portion of borax; and then add 3 c. c. of alcohol. Light the mixture and note the result. What does the experiment show?

156. In a clean iron spoon heat some crystallized boracic acid. The crystals will melt, and if the heat be continued, the mass will become pasty and will swell up as the water is expelled. After all the water has been driven off by strong heat, the anhydride is left as a clear, viscous liquid, from which long threads may be drawn out by touching to the surface of the liquid the end of a stick or glass rod and then gently pulling away the stick with the matter which has adhered to it. Allow the fused mass to cool, and examine its appearance.

EXERCISE 27. — SOLUBILITY.

[Prepare a wash bottle and a couple of stirring rods. See models.]

Solubility in water.

1. Weigh 3 portions of 1 gram each of powdered Na_2SO_4 , CaSO_4 , and PbSO_4 . Take three test tubes and place in each 10 c. c. of water. Into one of the tubes pour one portion of Na_2SO_4 and shake, and, if this dissolves, add the second, and if the second dissolves, add the third. Repeat the process with the CaSO_4 and PbSO_4 in the other tubes. If, however, the first lot fails to dissolve, ascertain whether any dissolves by filtering (see Appendix, § 15) some of the mixture *very* carefully and evaporating a few drops of the *filtrate* on a clean slip of platinum foil.

Solubility in hot and cold water.

2. Weigh 4 portions of 1 gram each of powdered BaN_2O_6 . Place 10 c. c. of water in a test tube and add one portion and shake. Note the result. Warm slowly, and as often as the salt is entirely dissolved add a new portion of 1 gram. Finally, bring the liquid to boiling. What does the experiment show?

Comparison of solubility in water.

3. [The salts *must* all be powdered!] Take 5 test tubes and place in each 10 c. c. of water. Into a test tube put 12 or 15 grams of potassium carbonate (K_2CO_3). Weigh the tube and contents and record the weight. From this test tube pour successive small portions of K_2CO_3 into one of the test tubes containing water as long as it will dissolve. Again weigh the test tube and contents. The loss in weight will be the weight of the K_2CO_3 dissolved in the water. Calculate the number of parts of the salt which have dissolved in 100 parts of water. In like manner test the solubility of KNO_3 , K_2SO_4 , SrSO_4 , and BaSO_4 , starting with 5 grams of the first, 3 grams of the second, and 1 gram of the third and fourth. Bring the results of the experiment into the form of a table. (See blackboard.)

4. [For students who are doing extra work.] Measure and pour into a flask 100 c. c. of water. Insert a thermometer in the flask by means of a perforated cork so that the temperature can be accurately observed. Note the temperature. Add Na_2SO_4 in weighed portions of 2 grams each until no more can be dissolved after thorough shaking, and make note of the amount required. Raise the temperature by means of a lamp underneath to 15° , remove the lamp and then add portions of 2 grams of Na_2SO_4 until you have determined the amount soluble at this tempera-

ture. Now raise to 20° and repeat the operation. Finally raise also to 25° , 30° , 35° , 40° . Make careful note of the amount added at each point and the total amount in solution at each temperature. Plot a curve of solubility upon a piece of cross section paper similar to the example upon the blackboard. This curve must be handed to the professor before the next exercise.

EXERCISE 28. — SOLUBILITY.

State clearly what is taught by each of the experiments which follow.

Use of different solvents.

5. Compare the solubility of bone ash in H_2O , dilute HCl , and dilute H_2SO_4 .

6. Compare the solubility of iodine in water, alcohol, and carbon disulphide. Apply no heat. In such cases as this it is advisable to take exceedingly small quantities of iodine and drop them into about 3 c. c. of the liquid; and watch the rapidity with which the iodine disappears, and judge from this in which liquid it is the most soluble.

7. Compare the solubility of NaCl in H_2O , strong HCl and alcohol.

Solubility in Mixtures.

8. Attempt to dissolve 1 gram of BaCl_2 in 3 c. c. of strong HCl . Finally add 10 c. c. of water.

9. Dissolve 2 grams of Na_2SO_4 in 4 c. c. of water, and add an equal volume of alcohol.

10. Dissolve 0.5 gram iodine in 2 c. c. alcohol and add 5 c. c. of water.

Neutralization of the solvent.

11. Take some calcium phosphate, dissolve in warm dilute HCl , and then add ammonia to alkaline reaction. Repeat the experiment, using oxalate of barium.

12. Dissolve a little AgCl in ammonia water and add dilute HNO_3 to acid reaction.

Absorption and development of heat.

13. Place 25 c. c. of strong HCl in a beaker. Note the temperature of the acid; dissolve 20 grams of Glauber's salt in the acid, and again note the temperature.

14. Place 10 c. c. of water in a small beaker. Note the temperature. Dissolve 10 grams of KOH in the water and again note the temperature.

EXERCISE 29. — SOLUBILITY.

Saturation.

15. Heat together 10 grams of alum and 8 c. c. of water, and allow to cool. Pour off the clear liquid and boil it for a few moments, and allow to cool again.

16. Place 10 grams of sodic acetate in a test tube, add 10 c. c. of water, and warm until the sodic acetate has entirely dissolved. Place a little cotton-wool in the mouth of the tube, set it aside, and allow to cool perfectly without moving. The liquid should be clear. Now drop into the liquid a small crystal of sodic acetate and observe the effect.

26. See exercise 30. Begin the experiment at this place.

Solution of liquids.

17. Test the solubility of the following liquids in water: alcohol, ether, olive oil, glycerine, carbon bisulphide. Proceed in each case as follows: Take 5 c. c. of water in a *clean* test tube; pour 1 c. c. of the liquid to be tested upon the water in the tube. Shake several times, and then observe the depth of the liquid layer above or below the water, if any such layer there be. Ether and carbon bisulphide are very inflammable and very volatile, and *must not be brought anywhere near a flame*.

18. Test the solubility of benzol in water and alcohol.

Physical and chemical solution.

19. Take two portions of 5 grams each of sal-soda. Dissolve one portion in 10 c. c. of water, evaporate to dryness slowly, and compare the substance obtained with the original salt in appearance, crystalline form, and taste. Dissolve the second portion in dilute chlorhydric acid, evaporate and compare.

20. Place 5 c. c. of alcohol in a test tube and add 1 c. c. of HCl, and shake. Drop a small piece of fused potassium carbonate into the tube. Place 5 c. c. of water in a test tube and add 1 c. c. of HCl. Drop a small piece of fused potassium carbonate into this tube. Explain.

21. Repeat Expt. 20, but use marble instead of potassium carbonate.

EXERCISE 30. — CRYSTALLIZATION.

Crystallization by solution.

22. Put about 0.5 gram PbCl_2 into a test tube with some 10 c. c. of water. Boil for a minute or two, and then filter quickly but carefully, receiving the filtrate in a clean test tube kept warm by being immersed in a beaker of hot water. When the liquid has filtered through, remove the test tube from the hot water and allow to cool. Examine the product under a microscope.

23. Place 5 grams of oxalic acid in a test tube, and add 10 c. c. of water. Heat until the acid has dissolved, and then allow to cool.

Crystallization by sublimation.

24. Heat a small lump of benzoic acid gently in a dry test tube. Repeat Expts. 36 and 62.

Crystallization by precipitation.

25. To a few drops of concentrated common salt solution add 5 c. c. of alcohol. Examine the precipitate under the glass.

Crystallization by solution and evaporation.

26. Place upon small watch glasses 1 c. c. each of solutions of potassium nitrate, potassium chlorate, potassium chromate, mercuric chloride, and sodium acetate, and allow the glasses to stand in your desk until the next exercise. Then observe carefully with the lens the forms of crystals obtained.

Purification by crystallization.

27. Weigh 25 grams of soda ash. Place in a beaker, and pour upon the ash 50 c. c. of water. Heat until the ash has dissolved, filter the turbid solution while hot, and allow to cool. Remove some of the crystals, place in an evaporating dish, and heat until they are reduced to a fine powder. Compare this powder with the original ash.

Water of Crystallization.

28. Grind together in a mortar 4 grams of crystallized sodium sulphate (Glauber's salt) and 2 grams of potassium carbonate.

29. Heat a crystal of copper sulphate in a test tube until no more steam passes off. Also repeat Expt. 9. What is taught? Heat a crystal of common salt in a test tube. How does it differ from the former case? Why?

EXERCISE 31. — SODIUM AND AMMONIUM SALTS.

157. Cover the bottom of a large bottle (at least a liter bottle) with hot water, drop in a piece of sodium as large as a small pea, and immediately cover the mouth of the bottle with a card or glass plate. Test the liquid with litmus paper. Also perform the experiment using cold water.

158. Place a bit of sodium upon charcoal, and ignite by means of the blow-pipe.

159. Wrap a bit of sodium in tissue paper and lay it upon a piece of ice.

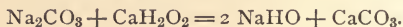
160. Place in a test tube 5 c.c. of concentrated NaOH, and add *slowly* 5 c.c. of strong HCl. Write the equation.

161. Weigh out exactly 9 grams of fine salt, and add it to 25 c.c. (measured) of water at the ordinary temperature. Shake until the salt has dissolved, then add another gram. If this fails to dissolve, bring the mixture to boiling. If all dissolves, show the solution.

162. Heat a crystal of NaCl upon platinum foil. State what action takes place and why. Observe the color of the flame due to the presence of sodium.

163. Mix 1 gram of powdered anhydrous Na_2SO_4 with an equal weight of powdered charcoal. Add a few drops of water and make a paste. Heat a portion of this paste before the blow-pipe on charcoal as strongly as possible. The Na_2SO_4 will be reduced to Na_2S . Treat a piece of the fused mass with dilute HCl. Also place a bit upon a silver coin and moisten with a drop of water.

164. *Soda by lime.* Dissolve 15 grams of crystallized sal-soda in 60 c.c. of water. Mix 4 grams of slaked lime with 15 c.c. of water. Heat the solution of sal-soda in an iron dish nearly to boiling, and add slowly the milk of lime, and boil for a few moments.



By decantation and evaporation the liquor obtained will yield NaOH. Pour the liquid from the iron dish into a beaker, allow to settle, decant a portion of the clear liquor back into the iron dish, and evaporate until the NaOH separates.

165. To a few cubic centimeters of a solution of ammonium chloride

in a test tube, add a few drops of a solution of caustic soda, and boil the liquid. Observe the odor.

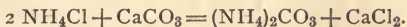
What similar experiment has already been performed?

166. Place a bit of sal-ammoniac in the bottom of a dry test tube and heat it slowly. Place bits of litmus paper at different positions in the test tube, and notice the action. Ask for explanation.

EXERCISE 32. — AMMONIUM SALTS AND POTASSIUM.

[Those who did not perform experiments 165 and 166 will do so before proceeding.]

167. Mix thoroughly 5 grams of NH_4Cl and 10 grams of CaCO_3 by grinding in a mortar, and heat in a small evaporating dish upon a sand bath under the hood. As soon as white fumes begin to rise place a large dry bottle or funnel over the dish and collect the white sublimate. Test this for a carbonate.



While the experiment is going on proceed with other experiments.

168. Place 100 grams of wood ashes on a filter and pour hot water over them, collecting the filtrate in a bottle and returning it upon the ashes two or three times, in order to obtain a strong solution. To exhaust the ashes of their potash they must, of course, be treated with successive portions of hot water. Test the solution with litmus paper and then evaporate it to dryness while succeeding experiments are being performed. Finally weigh the residue obtained and calculate the per cent of soluble matter in the ash. Treat the residue with HCl after the weight has been recorded.

169. Heat in a dry test tube strongly 3 grams of potassium tartrate. When the tube has become cool pour in boiling water, shake and filter. Add acid to the filtrate and notice that CO_2 is given off. This illustrates the formation of K_2CO_3 in the ashes.

170. Throw a piece of potassium, as large as a small pea, upon some cold water in the bottom of a large bottle, and place a card or glass plate over the mouth of the bottle. Test the liquid with litmus paper.

171. To a gas bottle in which carbonic acid is being steadily evolved according to Expt. 136, attach a chloride of calcium tube, and beyond

this drying tube a short tube of hard glass, from which an exit tube leads into a small open bottle. When the extinction of a lighted match in the open bottle proves the apparatus to be full of carbonic acid, thrust into the hard glass tube a bit of potassium as big as a pea, previously dried between folds of blotting paper; then gently heat the potassium with a lamp. When the tube has become cold, place it in a tube or bottle of water, filter the solution, note the appearance of the particles in the filter, evaporate the filtrate to dryness, add a few drops of dilute HCl.

172. *Test for potassium.* 1. Dip a platinum wire in a solution of KCl, and place the wire in the flame of a Bunsen burner. 2. To 1 c. c. of a solution of KCl add 1 drop of PtCl_4 . 3. Place in a test tube 3 c. c. of KCl solution, add an equal volume of a solution of tartaric acid, and shake.

EXERCISE 33.—BARIUM, CALCIUM, AND STRONTIUM.

At this exercise each student will have an opportunity to examine the spectroscope.

173. By means of iron wire, suspend three small bullets of well-burned coke from a ring of the iron stand. Heat the fragments in turn with the flame of the gas lamp, and observe the slightly yellowish flame which will be produced in each case; then moisten one of the pieces of coke with a solution of calcium chloride, the second with a solution of barium nitrate, the third with a solution of strontium nitrate, and again heat them in turn with the gas flame and observe the colors of the flames.

174. Mix carefully 3 grams of powdered KClO_3 , 5 grams of dry SrN_2O_6 and 2 grams of flowers of sulphur. Place the mixture upon an iron plate and ignite under the hood.

175. Repeat Expt. 174 but use 3 grams KClO_3 , 5 grams BaN_2O_6 , 1 gram of sulphur, and 0.5 gram powdered charcoal.

176. Pour a few drops of BaH_2O_2 solution upon a watch glass. Notice how quickly it absorbs CO_2 from the air.

177. Prepare a solution of "bicarbonate" of calcium by passing CO_2 into lime water (dilute) until clear. Boil a part of the solution and observe; to another portion add Na_2CO_3 solution, and observe.

178. Mix together 5 c. c. of a concentrated solution of CaCl_2 and 5 c. c. of a saturated solution of Na_2SO_4 . Examine the precipitate under the microscope. Write the equation.

179. Place a small piece of CaCl_2 upon a watch glass. Notice its condition at the end of the exercise. Illustrates deliquescence.

180. Into each of two test tubes of equal size put the same amount, say six drops (not more), of a CaCl_2 solution, marked "For Expt. 180." Into one of the tubes put 15 c. c. of NH_4Cl solution, into the other put 15 c. c. of water. Then add an equal quantity, say 5 c. c. of carbonate of ammonium to each. Show the result.

181. Fill an ignition tube one third full of bleaching powder, and arrange the apparatus so that any gas which is evolved may be collected over water. Heat the tube and test the gas collected

EXERCISE 34. — MAGNESIUM, CADMIUM AND ZINC.

182. Pour 5 c. c. of MgSO_4 solution into each of two test tubes. To the first add a few drops NH_4OH , to the second add 5 c. c. NH_4Cl and then a few drops of NH_4OH . Repeat the experiment, but use $(\text{NH}_4)_2\text{CO}_3$ instead of NH_4OH . Explain.

183. Place 3 c. c. of MgSO_4 solution in a test tube. Add 5 c. c. NH_4Cl and 5 c. c. NH_4OH . Dilute with an equal volume of water, add 3 c. c. Na_2HPO_4 solution and allow to stand. The crystalline precipitate thus obtained is the test for magnesium.

184. Take 10 c. c. of cadmium sulphate solution in a beaker, and dilute with 20 c. c. of water and add a few drops of HCl . Saturate with H_2S . Observe the appearance of the precipitate. Test its solubility in dilute HCl and dilute H_2SO_4 , also in NaHS .

185. Place a bit of zinc in each of three test tubes. To the first add dilute HNO_3 , to the second dilute HCl , and to the third dilute H_2SO_4 , and warm.

186. Place in a test tube 2 grams of zinc dust; pour upon it 5 c. c. of caustic soda solution, and warm the mixture. Explain the results.

187. Place 5 c. c. of zinc sulphate solution in a test tube, and add to it caustic soda solution in small quantity—say three or four drops. Finally add a larger quantity—say 3 c. c. Explain the results.

188. Immerse a piece of zinc in a solution of copper sulphate.

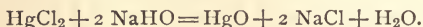
189. Place in each of two test tubes 5 c. c. of ZnSO_4 solution, dilute with an equal volume of water, and to one add 1 c. c. of dilute HCl and to the other 1 c. c. of acetic acid. Now saturate both tubes with H_2S and state what happens and why.

190. Heat 2 grams of ZnO in a dry test tube nearly to redness and observe the color. Allow to cool and notice again.

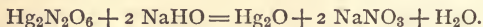
EXERCISE 35. — MERCURY.

191. Examine the action of dilute acids upon mercury as usual, but use a very small quantity of the metal.

192. To a solution of mercuric chloride add a solution of caustic soda as long as a precipitate falls.



193. To a solution of mercurous nitrate, add in the same way a solution of caustic soda.



194. Take 10 c. c. of HgCl_2 solution, dilute with 20 c. c. of H_2O , heat to boiling, and pass in H_2S to saturation, meanwhile observing changes in color. Filter, and test the solubility of the HgS in HCl , HNO_3 , and *aqua regia*.

195. Take two test tubes. Place in one 5 c. c. of $\text{Hg}_2\text{N}_2\text{O}_6$, and in the other 5 c. c. of HgN_2O_6 . Add HCl slowly, and notice the difference in the result. Explain.

196. Compare the solubility in hot and cold water of HgCl_2 and Hg_2Cl_2 .

197. To a solution of albumin add a few drops of corrosive sublimate. See § 497, p. 279 of Eliot and Storer's Manual.

198. Make a solution of ammonia to contain 1 part NH_3 by weight in 500,000 parts of water. See blackboard. Take two test tubes of equal diameter and place in one 25 c. c. of the dilute ammonia and in the other 25 c. c. of distilled water. Add a few drops of Nessler's solution (a solution of HgI_2 in KI and KOH) to each tube and allow to stand for five minutes. Notice the strong yellow color imparted to the solution containing the NH_3 . This is the best test for NH_3 when present in minute quantities.

199. Take 5 c. c. of HgCl_2 solution and add 10 drops of KI . Filter the precipitate and dry. Into a clean and *perfectly dry* test tube put a

small amount of the HgI_2 . Warm the middle portion of the tube until you cannot quite bear your hand upon it; then heat rather gently the lower part of the tube where the HgI_2 is. Explain.

EXERCISE 36. — COPPER.

200. Examine the action of dilute acids upon copper in the usual manner.

201. Mix equal weights of powdered CuCl_2 and Na_2CO_3 , and reduce upon charcoal.

202. Bind a bright copper coin with wire, in such manner that a strip of wire 8 or 10 c. m. long shall be left projecting from the coin; thrust the free end of the wire into a long cork or bit of wood, and by means of this handle hold the coin obliquely in a small flame of the gas lamp. Thrust the hot coin into water, and observe that it is at this stage covered with a red coating of copper suboxide. Replace the coin in the lamp and hold it in the hot oxidizing portion of the flame; it will soon become black from the formation of copper protoxide. After a rather thick coating of oxide has been formed, again quench the coin in water. Note what happens.

203. Dissolve a gram of copper in as small a quantity as possible of HNO_3 diluted with an equal bulk of water. Evaporate the solution to dryness; and finally ignite the residue until red fumes are no longer given off.

204. Place in a test tube, or small bottle, 8 or 10 c. c. of a cold dilute solution of copper sulphate, and add to it enough of a solution of caustic soda to render the mixture alkaline to test paper.

205. Repeat Expt. 204, with the difference that the solutions of caustic soda and copper sulphate are both heated to boiling, and are mixed while hot.

206. Again repeat Expt. 204, but instead of soda lye add to the copper salt ammonia water, drop by drop, and shake the tube after each addition of the ammonia.

207. To a cold dilute solution of copper sulphate add a few drops of a solution of grape sugar; then add enough caustic potash solution to dissolve the precipitate which forms at first. Warm the mixture; a yellowish precipitate of cuprous hydrate forms in the liquid, and by further heating is converted into the red cuprous oxide. Other reducing agents,

such as arsenious acid, for example, may be substituted for the grape sugar.

208. Acidify 10 c. c. of copper sulphate solution with acetic acid. Add a few drops of potassium ferrocyanide. This test for copper is even more delicate than the one given in Expt. 206.

209. Take 10 c. c. of CuSO_4 solution and saturate with H_2S . Filter and examine the solubility of the CuS in HCl , HNO_3 and Na_2Sx .

210. Prepare a little moist CuS , collect on a filter and leave for a day or two. Then put paper and all into a test tube, shake up with say 10 c. c. of water, let stand for a few minutes, filter, and add ammonia water to the filtrate. Explain the results obtained.

EXERCISE 37. — SILVER.

[Finish Expt. 210 if you began it.]

211. Place one or two dimes in a small flask, and cover them with nitric acid diluted with twice its bulk of water. Warm the flask gently in a place where there is a good draught of air; add more nitric acid, from time to time, if necessary to complete the solution, but carefully avoid having a great excess of the acid. Note the character of the evolved fumes and the color of the solution. Dilute the solution with an equal volume of water, place in it two or three copper coins and leave until the next exercise. Then collect the little plates of pure silver, which have separated from the solution, upon a filter, and wash them, first with water, and then with ammonia water, until the ammonia water no longer shows any tinge of blue. This silver washed finally with water and dried, is well-nigh pure; if it be again dissolved in nitric acid, the solution will contain nearly pure silver nitrate.

212. Fill three test tubes one third full of water, and pour into each a few drops of a moderately strong solution of silver nitrate. Add to the first test tube 2 or 3 c. c. of a solution of sodium chloride, to the second tube 2 or 3 c. c. of a solution of potassium bromide, and to the third tube 1 or 2 c. c. of a solution of potassium iodide; shake the mixture in each case and describe the precipitates.*

Withdraw from each test tube a portion of the precipitate it contains, and try to dissolve each precipitate in dilute nitric acid.

*Save all the silver residues.

Withdraw from each test tube another portion of the precipitate it contains, and treat each precipitate with ammonia water. Lastly, pour upon the remnants of the original precipitates in the three test tubes a solution of sodium hyposulphite.

213. Precipitate some curdy silver chloride by adding sodium chloride solution, or chlorhydric acid, to a solution of silver nitrate, so long as any precipitate is produced. Throw the precipitate upon a filter, and wash it with water; then open the filter, spread the chloride evenly over it, and place it in direct sunlight.*

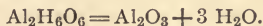
214. (1) To a solution of AgNO_3 add NaOH . (2) Pass H_2S through a solution of AgNO_3 . (3) Take three test tubes and place 2 c. c. of AgNO_3 solution in each. To the first add a few drops of Na_2HPO_4 , to the second a few drops of Na_2CO_3 , and to the third a few drops of K_2CrO_4 . Write the reactions.*

215. Precipitate AgCl as in Expt. 213, filter, dry and mix a portion or the whole with four times its bulk of carbonate of sodium and reduce upon charcoal.*

EXERCISE 38. — ALUMINUM.

216. Place a piece of aluminum in each of three test tubes and examine the action of acids upon the metal as in Expt. 185.

217. Precipitate a considerable quantity of $\text{Al}_2\text{H}_6\text{O}_6$ and filter. Place two thirds of the precipitate in a small porcelain dish and dry slowly over the lamp. Finally, when steam is no longer given off, heat strongly and observe the properties of the residue. Compare the solubility of the original precipitate and of the residue in HCl .



218. Ignite a small quantity of $\text{Al}_2\text{H}_6\text{O}_6$ before the blow-pipe upon charcoal. Moisten the white powder so obtained with CoN_2O_6 and continue the heating. Notice the blue color obtained. This is a test for aluminum.

219. Take a small quantity of a solution of cochineal, add to it an equal bulk of a solution of aluminum sulphate (or of common alum), and then add to the mixture ammonia water. A colored precipitate, consisting of aluminum hydrate and of the coloring matter of the cochineal, will be thrown down; it is the substance called carmine lake.

* Save all the silver residues.

220. Prepare an acetate of aluminum solution as follows: Dissolve 6 grams of sugar of lead (lead acetate) in 8 c. c. of hot water; also dissolve 8 grams of common alum in 12 c. c. of hot water; mix the two solutions and filter off the insoluble lead sulphate which is formed. In the solution thus prepared, soak a piece of cotton cloth, and then dry it. Treat this cloth, as well as a piece of ordinary cotton of the same size, with a solution of logwood, and observe the difference in the amount of color imparted to the fabric.

221. Place a considerable quantity of powdered aluminum sulphate in a test tube, add 10 c. c. of water and shake until a saturated solution is obtained. Take a second test tube and obtain a saturated solution of powdered K_2SO_4 . Now in a third test tube mix 5 c. c. each of these saturated solutions. Shake and observe the precipitate. Filter and examine the precipitate under the microscope and test its solubility in H_2O . Examine a piece of aluminum.

EXERCISE 39.—TIN, GOLD, AND PLATINUM.

222. Heat a piece of common tinned iron over the gas lamp until the tin has melted, thrust the plate into cold water in order that the tin may harden quickly, then remove the smooth surface of the metal by rubbing it first with a bit of paper moistened with dilute *aqua regia*, and then with paper wet with soda lye. Show the result.

223. Pour 20 c. c. of $SnCl_2$ solution into a beaker and then pour an equal volume of water upon it, but do not allow the two layers of liquid to mix. Thrust a slip of zinc through the liquid and observe what happens.

224. Examine the action of HCl , HNO_3 and H_2SO_4 upon metallic tin, both in dilute and concentrated state.

225. Dry some of the white powder obtained by the action of HNO_3 on tin, mix it with 2 or 3 times its bulk of powdered "binocalate of potash" and endeavor to reduce upon charcoal.

226. Take two test tubes and place in each 1 c. c. of $SnCl_2$ solution. Dilute with 10 c. c. of water. To one of the tubes add potassium permanganate until a faint color is obtained. Now saturate both tubes with H_2S . Explain. Test the solubility of the precipitates in HCl , concentrated and dilute, and Na_2S . Explain.

227. Take 1 c. c. of $SnCl_2$ solution and dilute with 10 c. c. of water. Add a few drops of $HgCl_2$. Give equation.

228. Place two drops of AuCl_3 in a beaker and dilute with 20 c. c. of water. To a few c. c. of SnCl_2 solution in a test tube add a few drops of Fe_2Cl_6 and then add a few drops of this mixture to the water containing AuCl_3 . Notice the coloration due to the formation of "purple of Cassius." This is the most delicate test for gold.

229. Pour 3 c. c. of a solution of ammonium chloride into a test tube, acidulate the liquid with chlorhydric acid, and add to it a drop of the solution of platinum chloride, and examine the precipitate under the microscope. Repeat the experiment, and this time take enough of the platinum solution and of the ammonium chloride to make half a teaspoonful of the yellow precipitate, taking care that at last there shall be a slight excess of free ammonium chloride rather than of platinum chloride in the supernatant liquid. Allow the precipitate to settle, separate it from the clear liquor by decantation, and dry it partially at a gentle heat. When the precipitate has acquired the consistence of slightly moistened earth, transfer it to a cup-shaped piece of platinum foil, and heat it to redness in the gas flame, as long as fumes of ammonium chloride continue to escape. All the chlorine, hydrogen and nitrogen will be driven off, and there will remain upon the foil a gray, loosely coherent, sponge-like mass of metallic platinum; it is called *platinum sponge*.

230. Hold the dry platinum sponge of Expt. 229 in a stream of hydrogen or of common illuminating gas issuing from a fine jet.

EXERCISE 40. — LEAD.

231. Heat a small fragment of lead upon charcoal in the oxidizing flame of the blow-pipe.

232. Fill a small ignition tube one fourth full of lead tartrate. Heat the tube gently until no more fumes are given off. Metallic lead will be left in the tube in a very finely divided condition. Cork tightly while hot and allow to become *perfectly* cold, then pour some of the powder into an evaporating dish, holding the tube high over the dish. Observe what happens.

233. Place a small piece of lead in each of three test tubes. Pour a few c. c. of dilute HNO_3 upon the first, a few c. c. of dilute HCl upon the second, and a few c. c. of dilute H_2SO_4 upon the third. Warm and observe the action.

234. Mix equal bulks of litharge and sodic carbonate and reduce upon charcoal.

235. Dissolve 0.5 gram lead acetate in 1 liter of water. Take 100 c. c. of this solution and dilute with about a liter of water; then take a small portion of this solution and pass H_2S through it. This is a most delicate test for Pb.

236. To a solution of PbN_2O_6 add HCl until no further precipitate is formed. Write the equation. Filter off the precipitate, and to one third of the filtrate add dilute H_2SO_4 ; to another third, add H_2S water; to the remainder, add K_2CrO_4 solution. Write equations. Dissolve the original precipitate in the least possible quantity of boiling water, filter and allow to cool.

EXERCISE 41. — CHROMIUM AND MANGANESE.

237. Weigh out 2 grams of chromic oxide, 4 grams of sodic carbonate and 2 grams of potassic nitrate. Mix thoroughly and fuse in an iron ladle until no more black specks are to be seen in the fused mass. Pour out the fused mass upon a broken piece of porcelain, allow to cool and break into small pieces. The fusion oxidized the chromium to the condition of chromic acid. The fused mass contains sodic chromate. Dissolve in water and add to a portion of the solution acetic acid to acid reaction and then acetate of lead. Acidify likewise two other portions, and to one add ZnCl_2 and to the other BaCl_2 .

238. To 10 c. c. of chrome alum solution add NH_4OH in excess. Filter the precipitate and ignite a portion in a porcelain crucible. Test the solubility of the original and of the ignited precipitate in HCl .

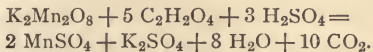
239. Place 20 c. c. of a saturated solution of bichromate of potassium in a beaker surrounded by cold water. Add slowly 25 c. c. of strong H_2SO_4 . Observe the deposition of chromic anhydride. Remove some of the crystals and test their solubility in water.

240. To a solution of MnCl_2 or MnSO_4 add NH_4HS . Collect the precipitate on a filter and allow to remain exposed to the air for some time.

241. Fuse together upon platinum foil 0.5 gram dry Na_2CO_3 , 0.1 gram KNO_3 and a few milligrams of MnO_2 . The color given to the fused mass is one of the best tests for manganese.

242. Place in a test tube 10 c. c. of dilute HNO_3 and 0.5 gram PbO_2 . Add a few milligrams of MnO_2 and boil. Allow to settle and observe; ask for an explanation of what you have observed.

243. In a beaker or flask dissolve 0.25 gram of crystallized oxalic acid in 50 c. c. of water, add 5 c. c. strong sulphuric acid, and warm the solution to about 60°. Then add a solution of potassium permanganate *drop by drop*, and observe that the color is at first immediately destroyed. Continue to add the permanganate until it is no longer decolorized. The reaction that has taken place may be thus represented:



The oxalic acid ($\text{C}_2\text{H}_2\text{O}_4$) is entirely converted into water and carbonic acid: the potassium permanganate gives up its oxygen and is converted into a mixture of manganese and potassium sulphates.

244. Mix together in an iron ladle 5 grams KOH, 3.5 grams powdered KClO_3 , 4 grams powdered MnO_2 and 10 c. c. water. Evaporate to dryness rapidly and then heat for a few moments until the mass has half fused, stirring constantly. The MnO_2 has been converted into potassium manganate. Treat with water, decant the clear green liquid and place 20 c. c. in an evaporating dish and notice any changes of color. Place another portion in a test tube and add a few drops of dilute H_2SO_4 . On account of these changes of color, potassium manganate is called chameleon mineral.

EXERCISE 42. — IRON.

245. Dissolve 2 or 3 small tacks in 8 or 10 c. c. of dilute sulphuric acid in a small beaker; when the evolution of hydrogen slackens, dilute with an equal bulk of water and filter into a small flask. To the liquid add a few drops of strong nitric acid, and heat it to boiling. The liquor will soon be colored dark brown by the nitrous fumes resulting from the decomposition of the nitric acid, which are for a short time held dissolved by the liquid; but this deep coloration soon passes away, and there is left only the yellowish red color of the ferric sulphate which has been formed. Add to the solution ammonia water, until the odor of the latter persists after agitation, and collect upon a filter the flocculent red precipitate of ferric hydrate.

246. Repeat Expt. 217 but make use of the $\text{Fe}_2\text{H}_6\text{O}_6$ obtained in Expt. 245. Compare the solubility of Fe_2O_3 and $\text{Fe}_2\text{H}_6\text{O}_6$ in HCl.

247. Compare the actions of the dilute acids upon metallic iron.

248. Pour a solution of copperas into an open capsule, and leave it exposed to the air for a day or two; the solution will gradually become yellow as the oxidation proceeds, and after a while a rusty precipitate of ferric oxide, or of highly basic ferric sulphate, will fall.

249. Place 20 c. c. of copperas solution in an evaporating dish and add ammonia water in excess. Observe the color of the precipitate. Allow to stand for half an hour, stirring frequently, and observe any change in the color of the precipitate. Write the equation.

250. Dip a small piece of cotton cloth in the solution of nutgalls, and allow it to become dry; then dip it in the solution of copperas and hang it up in damp air. Finally, try to wash out the color.

251. Dissolve one gram of copperas (iron sulphate) in 100 c. c. of water in a bottle of 200 c. c. capacity. Into the solution stir a mixture of 1 gram of finely powdered indigo and 1.5 grams of freshly slaked lime; fill up the bottle with water and cork it. Shake the bottle occasionally, and, at the next exercise, pour off, or remove with a pipette, a portion of the clear and nearly colorless liquid without disturbing the precipitate in the bottom of the bottle. Expose this liquid to the air in a shallow dish.

EXERCISE 43.—IRON, COBALT, AND NICKEL.

252. Dissolve a small crystal of copperas in water and add to the liquid a drop or two of a solution of ammonium sulphhydrate.

Finish 251.

253. To a solution of a ferric salt add a few drops of sulphocyanate of potassium and notice the color. This is a very delicate test for iron.

254. Dissolve 2 or 3 iron tacks in dilute HCl. Filter, and to a small portion of the filtrate add a few drops of potassium ferrocyanide solution. To a similar portion of the filtrate add a few drops of potassium ferricyanide. Notice the difference. These are tests for ferrous salts. Now to the remaining part of the filtrate add a few drops of HNO_3 and boil. Test small portions of this oxidized filtrate with ferro- and ferricyanide of potassium and show the results. If you have been successful in oxidizing the solution divide it into two portions. Through the first pass H_2S and note the effect. To the second add SnCl_2 and warm, and then test with ferro- and ferricyanide of potassium.

255. Prepare a borax bead, as will be shown. Place upon it a particle of any cobalt salt and heat in both reducing and oxidizing flame be-

fore the blow-pipe. Prepare a second bead and add a nickel salt. The colors obtained are good tests for the respective metals.

256. Write your name upon a piece of filter-paper, using CoCl_2 solution for ink. Allow to dry and observe that the writing is invisible. Now warm gently and observe the effect. Explain. Finally breathe upon the writing.

257. To 2 c. c. of CoN_2O_6 solution add an equal volume of dilute silicate of soda solution.

258. Pour 5 c. c. of a solution of any cobalt salt into a test tube and add 1 c. c. of acetic acid. Now add an equal volume of potassium nitrite solution. Allow to stand and observe the precipitate. It is a double nitrite of potassium and cobalt, and its formation is one of the best tests to show the presence of cobalt.

259. Take 10 drops or less of a solution of CoCl_2 . Dilute with water, then add ammonia water to alkaline reaction; if any precipitate appears, filter, and to the clear filtrate add a few drops of NH_4HS solution and note what happens. Now take the same amount of cobalt solution as before, and two teaspoonfuls of NH_4Cl solution, then add ammonia to alkaline reaction, note what happens and explain.

260. Heat gently in a dry test tube a few crystals of NiCl_2 .

261. Take two test tubes and place in one 2 c. c. of CoCl_2 and in the other 2 c. c. of NiCl_2 . Add to each NaOH to alkaline reaction, then a little KCN and then an equal volume of sodium hypochlorite and warm. The NiCl_2 gives a dark precipitate of $\text{Ni}_2\text{H}_6\text{O}_6$ while the CoCl_2 is unaffected. The reaction is often used to distinguish nickel in the presence of cobalt.



EXERCISE 44. — ARSENIC.

[Use great caution in performing all the experiments in this exercise.]

262. Place a few particles of "arsenious acid" in an open tube of hard glass about 10 c. m. long and heat over the lamp, holding the tube in a sloping position. Examine the sublimate with a microscope.

263. Drop into the point of a drawn out tube of hard glass, No. 5, a morsel of arsenious oxide, and above it place a splinter of charcoal; heat the coal red hot in the lamp flame, and then volatilize the arsenious acid.

264. Throw a particle of arsenious acid upon a piece of red hot charcoal. Notice any peculiar odor.

265. [Under the Hood.] Arrange a hydrogen generator as in the model, having a delivery tube beyond the calcium chloride tube drawn down to a narrow bore in several places. Generate hydrogen from zinc and dilute chlorhydric acid, and when *pure* hydrogen escapes from the delivery tube light the jet. Into the generator through the thistle tube pour a  few drops  of a solution of arsenious acid in chlorhydric acid; watch the flame to see whether it appears to alter in character. Hold a concave bit of cold porcelain in the flame; a brownish black deposit of arsenic is formed; collect several of these spots and reserve them for future experiment. While the stream of arseniureted hydrogen is passing through the delivery tube, heat the tube with the flame of a Bunsen lamp, a little behind one of the constricted portions. The arseniureted hydrogen is decomposed, and arsenic is deposited in the tube as a metallic mirror. Produce several of these mirrors and save them, and then wash out the generator, zinc and all, into the sink under the small hood. [This is Marsh's test.]

266. Into a small bottle put several teaspoonfuls of a chlorhydric acid solution of arsenious acid, add water enough to nearly fill the bottle and allow H_2S to bubble through the liquid until the liquid smells strongly of sulphureted hydrogen. Collect the precipitate (what is it?) upon a filter; and test its solubility in HCl and Na_2Sx solution.

267. Take two test tubes and place in each 5 c. c. of arsenite of sodium. Make one strongly acid with HCl and the other strongly alkaline with $NaOH$. Pass H_2S into each tube and note the result. Explain. Acidulate with HCl the one that is alkaline.

268. Take two test tubes. Place in one 5 c. c. of arsenite of sodium, in the other 5 c. c. of arseniate of sodium. Acidify with HCl and pass in H_2S . Explain.

269. Place in one test tube 5 c. c. of arsenite of sodium, and in another 5 c. c. of arseniate of sodium and add a few drops of $AgNO_3$ to each. This difference is the distinguishing test between the two acids and their salts. Save the silver residues.

EXERCISE 45. -- ANTIMONY AND BISMUTH.

270. [Under the hood.] In a flask of about 200 c. c. capacity, heat gently 0.5 gram of finely powdered antimony with 30 c. c. of strong chlorhydric acid, to which ten drops of nitric acid have been added. When complete solution has been effected, evaporate to less than one half its bulk; pour a little of the chloride into water. Evaporate the rest of the solution to the consistency of a thick syrup; it is the butter of antimony.

271. Pour some of the antimony terchloride into a small bottle nearly full of water, and then add just enough chlorhydric acid to dissolve the white precipitate which is formed. Through the clear solution pass a stream of hydrogen sulphide. Filter and test the solubility of Sb_2S_3 in dilute HCl , in hot strong HCl , and in Na_2Sx .

272. Perform precisely as 265, except that a solution of an antimony compound is to be substituted for the solution containing arsenic. Reserve the mirrors as in Expt. 265.

273. Compare together the spots obtained on porcelain from arseniureted hydrogen (Expt. 265) and from antimoniuireted hydrogen (Expt. 272). 1. The arsenical spot has a metallic luster and a brown color when thin; the stain of antimony has a feeble luster, and is smoky black. 2. The arsenical stain disappears readily on the application of a heat below redness; the stain of antimony is volatile only at a red heat. On account of the comparative want of volatility which characterizes the antimony deposit, the mirrors of antimony obtained in the glass tube are always deposited nearer the heated portion of the tube than the arsenic mirrors are. 3. The arsenical stains may be distinguished, moreover, from the antimonial stains by means of "chloride of lime," which immediately dissolves arsenical spots, but leaves antimonial spots unaffected for a long time. 4. An antimony stain will dissolve readily in a few drops of a solution of sulphhydrate of ammonium which has become yellow by keeping; when such a solution is evaporated to dryness, a bright orange stain remains. The arsenical stain, on the contrary, is not perceptibly affected by the yellow sulphhydrate of ammonium solution, unless heat is applied.

274. Connect the tube of hard glass in which the arsenic mirrors were formed, in Expt. 265, with a sulphureted hydrogen generator, interposing between the tube and the generator a suitable drying-tube or bottle filled with calcium chloride; then transmit through the tube a *very slow*

stream of hydrogen sulphide gas, and heat the mirrors with a small gas flame, proceeding from the outer to the inner border of the mirrors in the direction opposite to that of the gas current.

Repeat the same process with the tube containing the antimony mirrors obtained in Expt. 272.

275. Place 5 c. c. of tartar emetic (tartrate of antimony and potassium) in each of two test tubes, and make one slightly acid with HCl and the other strongly alkaline with NaOH, and saturate with H_2S . Explain.

276. Repeat Expt. 270 but use bismuth.

277. Repeat Expt. 275 but use BiCl_3 instead of tartar emetic.

278. Weigh out 15 grams of bismuth, 8 grams of lead and 8 grams of tin. Heat a beaker of water to boiling, and convince yourself that neither the bismuth, lead or tin will melt in the hot water. Now fuse the metals together in an iron spoon. Allow the mass to cool and place it in a beaker of boiling water. This alloy is known as fusible metal. Pour a little of the fused metal into a narrow test tube and allow to cool. Explain its action.

EXERCISE 46. — CHEMICAL CHANGES.

Analyze the following reactions, and tell whether they are analytical, synthetical, or metathetical. Also explain any modifications from the action which would be naturally expected.

1. To 0.5 c. c. of sodic carbonate solution add dilute HCl.
2. Place a bit of copper in a test tube and add dilute HNO_3 .
3. Repeat Expt. 88. Test, before warming, for SO_2 , and again after warming.
4. Mix 2 grams NH_4Cl and 2 grams CaH_2O_2 . Place in a dry test tube and test for NH_3 . Now warm and repeat the test.
5. To 5 c. c. of sodium hyposulphite solution add dilute HCl. To a second 5 c. c. add acetic acid.
6. Place 5 c. c. of CuSO_4 solution in each of three test tubes. To one add an equal volume of NaCl solution, to another a few c. c. of BaCl_2 solution and compare the three tubes.
7. Shake a few grams of CaSO_4 with water, filter and add $(\text{NH}_4)_2\text{CO}_3$.

8. Take 2 c. c. of a solution of As_2O_3 in HCl and dilute to 10 c. c. with water. Add $(\text{NH}_4)_2\text{Sx}$ drop by drop at first, finally a large excess and boil. Now acidify with HCl .

9. Repeat the last experiment, but use CdCl_2 instead of As_2O_3 .

10. Place 3 c. c. of Fe_2Cl_6 in each of two test tubes and dilute with 20 c. c. of water. To one add a few drops of K_4FeCy_6 , to the other a few drops of KSCN .

11. Into a test tube put 10 c. c. of water. Add to it 3 drops of KSCN solution; then add one drop of $\text{Fe}_2(\text{NO}_3)_6$. Now add AgNO_3 solution drop by drop until there is a change of color. Explain. Save all silver residues.

12. To 5 c. c. of CuSO_4 solution add NH_4OH in excess.

13. Heat a mixture of mercuric sulphate and sodic chloride in a matrass.

14. Pass a current of CO_2 through a dilute solution of sodium silicate.

EXERCISES 47 AND 48. — PRINCIPLES OF QUALITATIVE ANALYSIS.

1. Take 1 c. c. of an aqueous solution of CuSO_4 and 1 c. c. of AgNO_3 solution. Add dilute HCl a few drops at a time; shake and allow to settle after each addition. When further addition of HCl produces no further precipitation (of what? Answer from past experience), collect the precipitate on a filter. To the filtrate add ammonia little by little to alkaline reaction and explain the observed result (also from past experience).

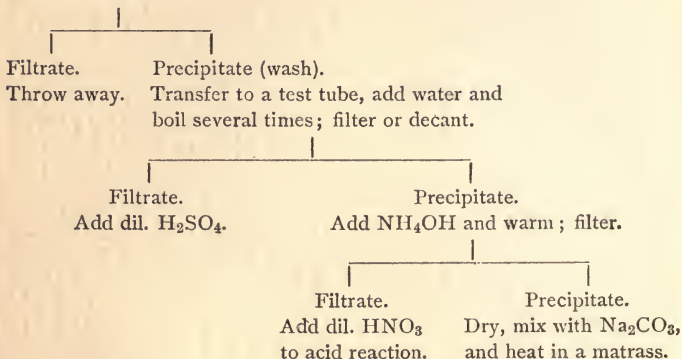
2. Take about 1 c. c. of each of the following solutions and add to each, in a test tube, a few drops of HCl (dilute): — AgNO_3 , PbN_2O_6 , CuN_2O_6 , $\text{Hg}_2\text{N}_2\text{O}_6$, HgN_2O_6 , FeSO_4 , ZnSO_4 , MgSO_4 , KNO_3 . Note in which cases a precipitate appears.

3. To 1 c. c. of a solution of AgNO_3 add 2 c. c. or so of water and then HCl (dilute) a little at a time with shaking until further addition produces no more precipitate. Wash by decantation. Boil one portion of precipitate with water; treat a second portion with ammonia water.

Repeat this procedure, using a solution of PbN_2O_6 instead of AgNO_3 , also using $\text{Hg}_2\text{N}_2\text{O}_6$. To small portions of solutions of AgNO_3 , PbN_2O_6 and $\text{Hg}_2\text{N}_2\text{O}_6$, add H_2SO_4 (dilute).

4. Mix together some of each of the three solutions (AgNO_3 , PbN_2O_6 , $\text{Hg}_2\text{N}_2\text{O}_6$), and proceed as indicated in the following scheme.

Add HCl and filter.



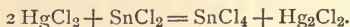
Then take an unknown solution which may contain compounds of one or more of the elements lead, silver or mercury (ous).

5. Three general methods by which the presence of a given element may be ascertained.

1. By isolating the element itself. Recall or perform, if you do not remember exactly what happened, Expts. 215, 234, 188.

2. By producing a characteristic compound of the element sought, as we have already proved the presence of Ag , Pb or $[\text{Hg}_2]$ "by the appearance and action of their chlorides."

3. By producing a precipitate not *containing the element* sought, but which could be formed only in presence of that element or some particular compound of it. Expt. 227. The precipitate is mercurous chloride (Hg_2Cl_2) and the HgCl_2 may be used as a test for tin.



6. Make a solution containing say 1 c. c. of each of the following solutions: Fe_2Cl_6 , ZnCl_2 , CaCl_2 , MgCl_2 , and a teaspoonful of NH_4Cl . Add ammonia until the odor persists after shaking, collect the precipitated $\text{Fe}_2\text{H}_6\text{O}_6$ on a filter, and to the filtrate add $(\text{NH}_4)\text{HS}$. Collect the

precipitated ZnS on a filter and to the filtrate add $(\text{NH}_4)_2\text{CO}_3$. Collect the precipitated CaCO_3 on a filter and to the filtrate add Na_2HPO_4 . The precipitate is a phosphate of Mg and NH_4 .

EXERCISES 49-53. — PREPARATIONS.

1. *Purification of Sal-Ammoniac.*

Place 100 grams of commercial NH_4Cl in a six-inch evaporating dish and add 300 c. c. of water. Heat and stir until the NH_4Cl has dissolved. Add a few c. c. of NH_4OH to precipitate any iron present, and filter while hot through a large filter. The filtrate should be colorless. Carefully wash the dish and return the filtrate to it and evaporate with continual stirring. Continue the evaporation not too rapidly until the NH_4Cl is obtained as a fine white powder free from lumps. Weigh.

2. *Glauber's Salt.*

Place 200 c. c. of water in a beaker which will hold 500 c. c. Weigh in a small beaker 20 grams of H_2SO_4 and pour this slowly into the 200 c. c. of water. Weigh 60 grams of sal-soda and dissolve by warming in the least possible quantity of water. Add the solution of sal-soda little by little to the dilute H_2SO_4 , carefully avoiding all loss by foaming. Test with litmus paper, and if the liquid is still acid, add sal-soda solution until it has become slightly alkaline. Evaporate in a large dish to one third of the original volume and filter while hot. Set the filtrate aside in the dish until the next exercise. Cover the dish with paper to protect it from dust. Pour off the supernatant liquid from the crystals when they are formed.

3. *Ammonium Iron Alum.*

Ferric Sulphate. — Weigh 30 grams of H_2SO_4 in a beaker and dilute with 15 c. c. of water. Stir thoroughly into the acid 20 grams of $\text{Fe}_2\text{H}_6\text{O}_6$. Add 20 c. c. of water, warm gently and stir frequently while the ammonium sulphate (see below) is being prepared. The $\text{Fe}_2\text{H}_6\text{O}_6$ will gradually dissolve. Finally dilute with water, filter and evaporate until the liquid becomes turbid from the separation of the ferric sulphate. Mix this solution with that of the ammonium sulphate prepared in the mean time, stir, cover with paper and set aside until the next exercise.

Ammonium Sulphate. — Weigh 10 grams of H_2SO_4 and place it in an evaporating dish. Dilute with 25 c. c. of water. Now add in small portions ammonia until a permanent smell of the same is obtained after stirring the liquid. Filter and evaporate to 15 c. c.

4. *Sugar of Lead.*

Place 20 grams of PbO in an evaporating dish, cover with water and reduce to a thin paste with a pestle. Add 45 grams of $\text{HO}(\text{C}_2\text{H}_3\text{O})$ and warm until the PbO has dissolved. Filter and evaporate until a drop of the liquid cooled upon a watch glass deposits numerous crystals. Cover and allow to stand.

5. *Barium Carbonate.*

Dissolve 18 grams of BaCl_2 in 150 c. c. of water by warming in a flask, filter and pour into a large bottle. Powder in a porcelain mortar some $(\text{NH}_4)_2\text{CO}_3$ and weigh out 6 grams. To this add 10 c. c. of ammonia water and then 10 c. c. of water, and stir without warming until the $(\text{NH}_4)_2\text{CO}_3$ is dissolved. Filter if necessary. Pour the solution of $(\text{NH}_4)_2\text{CO}_3$ into the solution of BaCl_2 , shake well and allow to settle. Pour a few c. c. of the liquid on the top into a test tube and add a little $(\text{NH}_4)_2\text{CO}_3$. If a precipitate is obtained, more $(\text{NH}_4)_2\text{CO}_3$ must be added to the BaCl_2 . Repeat the test until a small excess of $(\text{NH}_4)_2\text{CO}_3$ is present. Allow the precipitate to settle and wash by decantation four times. Test the last wash-water with AgNO_3 for chlorine. If chlorine is present in any considerable quantity, continue to wash by decantation until only a trace remains. Then filter, wash and dry the precipitate carefully, and give the BaCO_3 to the assistant.

6. *Chrome Yellow.*

Dissolve 25 grams of sugar of lead in 50 c. c. of water and filter. Pour the solution into a large bottle. Dissolve 10 grams of bichromate of potassium in 20 c. c. of water and pour this solution into the one of sugar of lead in the large bottle and shake. Add 100 c. c. of water and allow the precipitate to subside, and if the supernatant liquid is yellow add a further small quantity of sugar of lead solution and shake again. Continue this until the sugar of lead is present in slight excess. Fill the bottle full of water and wash the precipitate by decantation until the wash-water gives no further brown color when H_2S is passed through it. Now bring the precipitate upon a filter.

7. *Chrome Orange.*

Take one half of the precipitate obtained in the last experiment, place it in a small evaporating dish, add a few c. c. of water and rub carefully with a pestle until all the lumps are broken up. Dissolve 3 grams of NaOH in 10 c. c. of water and pour this solution upon the chrome yellow, stir constantly and keep at the boiling point for 10 minutes. Pour into the bottle, taking care to wash out all adhering chrome orange. Wash now by decantation until the wash-water gives no further test for an alkali with litmus paper. Filter, dry and weigh. Give both chrome yellow and chrome orange to assistant.

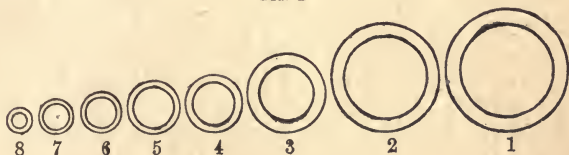


APPENDIX.

CHEMICAL MANIPULATION.

1. Glass-tubing. — Two qualities of glass-tubing are used in chemical experiments, — that which softens readily in the flame of a gas- or spirit-lamp, and that which fuses with extreme difficulty in the flame of the blast-lamp. These two qualities are distinguished by the terms *soft* and *hard* glass. Soft glass may be used for all purposes, except the intense heating, or ignition, of dry substances. Fig. I represents the most convenient sizes of glass-tubing, both hard and soft, and shows also the proper thickness of the glass walls for each size.

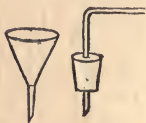
FIG. I.



2. Cutting and Cracking Glass. — Glass-tubing and glass-rod must generally be cut to the length required for any particular apparatus. A sharp triangular file is used for this purpose. The stick of tubing, or rod, to be cut is laid upon a table, and a deep scratch is made with the file at the place where the fracture is to be made. The stick is then grasped with the two hands, one on each side of the

mark, while the thumbs are brought together just at the scratch. By pushing with the thumbs and pulling in the opposite direction with the fingers, the stick is broken squarely at the scratch, just as a stick of candy or a dry twig may be broken. The sharp edges of the fracture should invariably be made smooth, either with a wet file, or by softening the end of the tube or rod in the lamp. (See Appendix, § 3.) Tubes or rods of sizes 4 to 8 inclusive may readily be cut in this manner; the larger sizes are divided with more difficulty, and it is often necessary to make the file-mark both long and deep. An even fracture is not always to be obtained with large tubes. The lower ends of glass funnels, and those ends of gas delivery-tubes which enter the bottle or flask in which the gas is generated, should be filed off, or

FIG. II.



ground off on a grindstone, obliquely (Fig. II), to facilitate the dropping of liquids from such extremities.

In order to cut glass plates, the glazier's diamond must be resorted to. For cutting exceedingly thin glass tubes and other glass ware, like flasks, retorts

and bottles, still other means are resorted to, based upon the sudden and unequal application of heat. The process divides itself into two parts, the producing of a crack in the required place, and the subsequent guiding of this crack in the desired direction. To produce a crack, a scratch must be made with the file, and to this scratch a pointed bit of red-hot charcoal, or the jet of flame produced by the mouth blowpipe, or a very fine gas-flame, or a red-hot glass-rod may be applied. If the heat does not produce a crack, a wet stick or file may be touched upon the hot spot. Upon any part of a glass surface except the edge, it is not possible to control perfectly the direction and extent of this first crack; at an edge a small crack may be started with tolerable certainty by carrying the file-mark entirely *over* the edge. To guide the crack thus started, a pointed bit of charcoal or slow-match may be used. The hot point must be kept on the glass from 1 c. m. to 0.5 c. m. in advance of the point of the crack. The crack will follow the hot point, and may therefore be carried in any desired direction. By turning and blowing upon the coal or slow-match, the point may be kept sufficiently hot. Whenever the place of experiment is supplied with common illuminating gas, a very small jet of burning gas may be advantageously substituted for the hot coal or slow match. To obtain such a sharp jet, a piece of hard glass tube, No. 5, 10 c. m. long, and drawn to a very fine point (see Ap-

pendix § 3), should be placed in the caoutchouc tube which ordinarily delivers the gas to the gas-lamp, and the gas should be lighted at the fine extremity. The burning jet should have a fine point, and should not exceed 1.5 c. m. in length. By a judicious use of these simple tools, broken tubes, beakers, flasks, retorts and bottles may often be made to yield very useful articles of apparatus. No sharp edges should be allowed to remain upon glass apparatus. The durability of the apparatus itself, and of the corks and caoutchouc stoppers and tubing used with it, will be much greater, if all sharp edges are removed with the file, or, still better, rounded in the lamp.

3. Bending and Closing Glass-tubes.—Tubing of sizes 5 to 8 inclusive can generally be worked in the common gas- or spirit-lamp; for larger tubes the blast-lamp is necessary (see Appendix, § 6). Glass tubing must not be introduced suddenly into the hottest part of the flame, lest it crack. Neither should a hot tube be taken from the flame and laid at once upon a cold surface. Gradual heating and gradual cooling are alike necessary, and are the more essential the thicker the glass; very thin glass will sometimes bear the most sudden changes of temperature, but thick glass and glass of uneven thickness absolutely require slow heating and annealing. When the end of a tube is to be heated, as in rounding sharp edges, more care is required in consequence of the great facility with which cracks start at an edge. A tube should, therefore, always be brought first into the current of hot air beyond the actual flame of the gas- or spirit-lamp, and there thoroughly warmed, before it is introduced into the flame itself. If a blast-lamp is employed, the tube may be warmed in the smoky flame, before the blast is turned on, and may subsequently be annealed in the same manner; the deposited soot will be burnt off in the first instance, and in the last, may be wiped off when the tube is cold. In heating a tube, whether for bending, drawing or closing, the tube must be *constantly* turned between the fingers, and also moved a little to the right and left, in order that it may be uniformly heated all around, and that the temperature of the neighboring parts may be duly raised. If a tube, or rod, is to be heated at any part but an end, it should be held between the thumb and first two fingers of each hand in such a manner that the hands shall be below the tube, or rod, with the palms upward, while the lamp-flame is between the hands. When the end of a tube, or rod, is to be heated it is best to begin by warming the tube, or rod, about 2 c. m. from the end, and from thence to proceed slowly to the end,

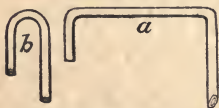
The best glass will not be blackened or discolored during heating. The blackening occurs in glass which, like ordinary flint glass, contains lead (silicate). Glass containing much lead is not well adapted for chemical uses. The blackening may sometimes be removed by putting the glass in the upper or outer part of the flame, where the reducing gases are consumed, and the air has the best access to the glass. The blackening may be altogether avoided by always keeping the glass in the oxidizing part of the flame.

Glass begins to soften and bend below a visible red heat. The condition of the glass is judged of as much by the fingers as the eye; the hands feel the yielding of the glass, either to bending, pushing or pulling, better than the eye can see the change of color or form. It may be bent as soon as it yields in the hands, but can be drawn out only when much hotter than this. Glass-tubing, however, should not be bent at too low a temperature; the curves made at too low a heat are apt to be flattened, of unequal thickness on the convex and concave sides, and brittle.

In bending tubing to make gas delivery-tubes and the like, attention should be paid to the following points: 1st, the glass should be equally hot on all sides; 2d, it should not be twisted, pulled out or pushed together during the heating; 3d, the bore of the tube at the bend should be kept round, and not altered in size; 4th, if two or more bends be made in the same piece of tubing (Fig. III, *a*), they should all be in the same plane, so that the finished tube will lie flat upon the level table.

When a tube or rod is to be bent or drawn close to its extremity, a temporary handle may be attached to it by softening the end of the tube, or rod, and pressing against the soft glass a fragment of glass tube, which will adhere strongly to the softened end. The handle may subsequently be removed by a slight blow, or by the aid of a file. If a considerable bend is to be made, so that the angle between the arms will be very small or nothing, as in a siphon, for example, the curvature can not be well produced at one place in the tube, but should

FIG. III.



be made by heating, progressively, several centimetres of the tube, and bending continuously from one end of the heated portion to the other (Fig. III, *b*). Small and thick tube may be bent more sharply than large or thin tube.

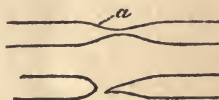
In order to draw a glass tube down to a finer bore, it is simply necessary to thoroughly soften on all sides one or two centimetres'

length of the tube, and then, taking the glass from the flame, to pull the parts asunder by a cautious movement of the hands. The larger the heated portion of glass, the longer will be the tube thus formed. Its length and fineness also increase with the rapidity of motion of the hands. If it is desirable that the finer tube should have thicker walls in proportion to its bore than the original tube, it is only necessary to keep the heated portion soft for two or three minutes before drawing out the tube, pressing the parts slightly together the while. By this process the glass will be thickened at the hot ring.

To obtain a tube closed at one end, it is best to take a piece of tubing, open at both ends, and long enough to make two closed tubes. In the middle of the tube a ring of glass, as narrow as possible, must be made thoroughly soft. The hands are then separated a little, to cause a contraction in diameter at the hot and soft part. The point of the flame must now be directed, not upon the narrowest part of the tube, but upon what is to be the bottom of the closed tube. This point is indicated by the line *a* in Fig. IV. By

FIG. IV.

withdrawing the right hand, the narrow part of the tube is attenuated, and finally melted off, leaving both halves of the original tube closed at one end, but not of the same form; the right-hand half is drawn out into a long point, the other is more roundly closed. It is not possible to close handsomely the two pieces at once. The tube is seldom perfectly finished by the operation; a superfluous knob of glass generally remains upon the end. If small, it may be got rid of by heating the whole end of the tube, and blowing moderately with the mouth into the open end. The knob being hotter, and therefore softer than any other part, yields to the pressure from within, spreads out and disappears. If the knob is large, it may be drawn off by sticking to it a fragment of tube, and then softening the glass above the junction. The same process may be applied to the too pointed end of the right-hand half of the original tube, or to any misshapen result of an unsuccessful attempt to close a tube, or to any bit of tube which is too short to make two closed tubes. When the closed end of a tube is too thin, it may be strengthened by keeping the whole end at a red heat for two or three minutes, turning the tube constantly between the fingers. It may be said in general of all the preceding operations before the lamp, that *success depends on keeping the tube to be heated in constant*



rotation, in order to secure a uniform temperature on all sides of the tube.

9. (Abridged.) **Corks.**—The best corks generally need to be softened before using; this may be effected by rolling the cork under a board upon a table, or under the foot upon a clean floor, or by gently squeezing it on all sides with a “cork-squeezer.”

In boring holes through corks to receive glass tubes use is made of a round file, and the aim is to make the hole as cylindrical as possible and not too large.

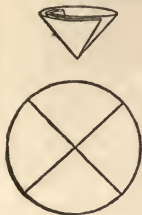
A flask which presents sharp or rough edges at the mouth can seldom be tightly corked, for the cork cannot be introduced into the neck without being cut or roughened; such sharp edges must be rounded in the lamp. In thrusting glass tubes through bored corks: (1.) The tube should be grasped very close to the cork, in order to escape cutting the hand which holds the cork, should the tube break. (2.) A thistle tube must never be held by the thistle in driving it through the cork, nor a bent tube grasped at the bend, unless the bend comes immediately above the cork. (3.) The tube must not be pushed straight into the cork, but screwed in, as it were, with a slow rotary as well as onward motion. Joints made with corks should always be tested before the apparatus is used by blowing into the apparatus and at the same time stopping up all legitimate outlets. Any leakage is revealed by the disappearance of the pressure created.

15. **Filtering.**—Filtration is resorted to in order to separate a finely divided solid from a liquid. The filter may be made of paper, cloth, tow, cotton, asbestos, and other substances. Paper is the substance oftenest used. A good filtering paper must be porous enough to filter rapidly, and yet sufficiently close in texture to retain the finest powders; and it must also be strong enough to bear, when wet, the pressure of the liquid which must be poured upon it.

Filtering paper is commonly sold in sheets, which may be cut into circles of any desired diameters for use, according to the various scales of operation and quantities of liquids to be filtered, or packages of “cut-filters” may be procured ready-made from the dealers in chemical ware.

There are two ready methods of preparing filters for use. According to the first method, shown in Fig. XXIV, a circle of paper is folded over on its own diameter, and the semicircle thus obtained is doubled once upon itself into the form of a quadrant; the paper thus folded is opened so that three thicknesses shall come upon one side,

FIG. XXIV.



and one thickness upon the other, as shown in the upper half of Fig. XXIV ; the filter is then placed in a glass funnel, the angle of which should be precisely that of the opened paper, viz., 60° . The paper may be so folded as to fit a funnel whose angle is more or less than 60° , but this is the most advantageous angle, and funnels should be selected with reference to their correctness in this respect.

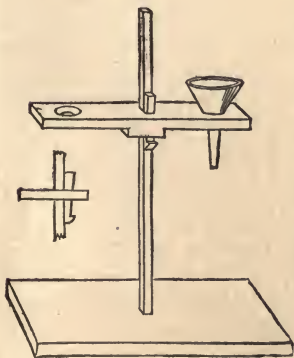
In the second method of folding filters, the circle of paper is doubled once upon itself as before into the form of a semicircle, and a fold equal to one quarter of this semicircle is turned down on each side of the paper. Each of the quarter semicircles is then folded

FIG. XXV.



back upon itself, as shown in the lower half of Fig. XXV ; the filter is opened, without disturbing the folded portions, and placed in the funnel. Filtration can be rapidly effected with this kind of filter, for the projecting folds keep open passages between the filter and the funnel, and thus facilitate the passage of the liquid. That portion of the circle of paper which must necessarily be folded up in order to give the requisite conical form to a paper filter retards filtration in the first manner of folding, but helps it in the second.

FIG. XXVI.



Coarse and rapid filtering can be effected with cloth bags ; also by plugging the neck of a funnel loosely with tow or cotton. If a very acid or very caustic liquid, which would destroy paper, cotton, tow or wool, is to be filtered, the best substances wherewith to plug the neck of the funnel are asbestos and gun-cotton, neither of which is attacked by such corrosive liquids.

FIG. XXVII.



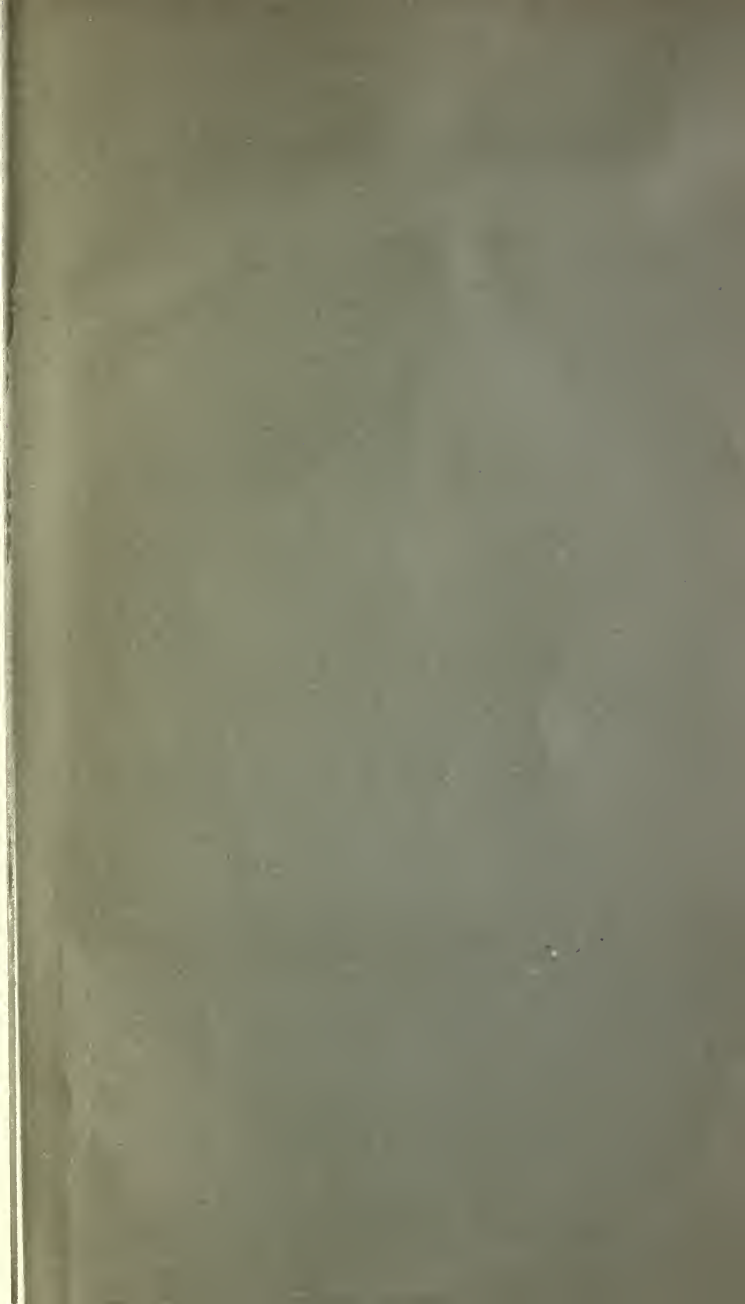
The glass funnel which holds the filter generally requires an independent support, for it is seldom judicious, or possible, to support

the funnel directly upon the vessel which receives the *filtrate*, as the clear liquid which runs through the filter is called. The iron stand (Fig. XVII) may be used for this purpose; and wooden stands, of the form represented in Fig. XXVI, adapted expressly for holding funnels, are very convenient and not expensive. In general, care should be taken that the lower end of the funnel touch the side or edge of the vessel into which the filtrate descends, in order that the liquid may not fall in drops, but run quietly down without splashing. Sometimes there is no objection to thrusting a funnel directly into the neck of a bottle or flask, but in this case an ample exit for the air in the bottle must be provided (Fig. XXVII).

[The "Appendix" is taken from Eliot and Storer's *Elementary Manual* by permission of the authors and publishers.]







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